PB80-188378

Coal Gasification/Gas Cleanup Test Facility
Volume I. Description and Operation

North Carolina State Univ. at Raleigh

Prepared for

Industrial Environmental Research Lab.
Research Triangle Park, NC

Mar 80

U.S. Department of Commerce
National Technical Information Service
NTIS

PB80-188378

United States
Environmental Protection
Agency

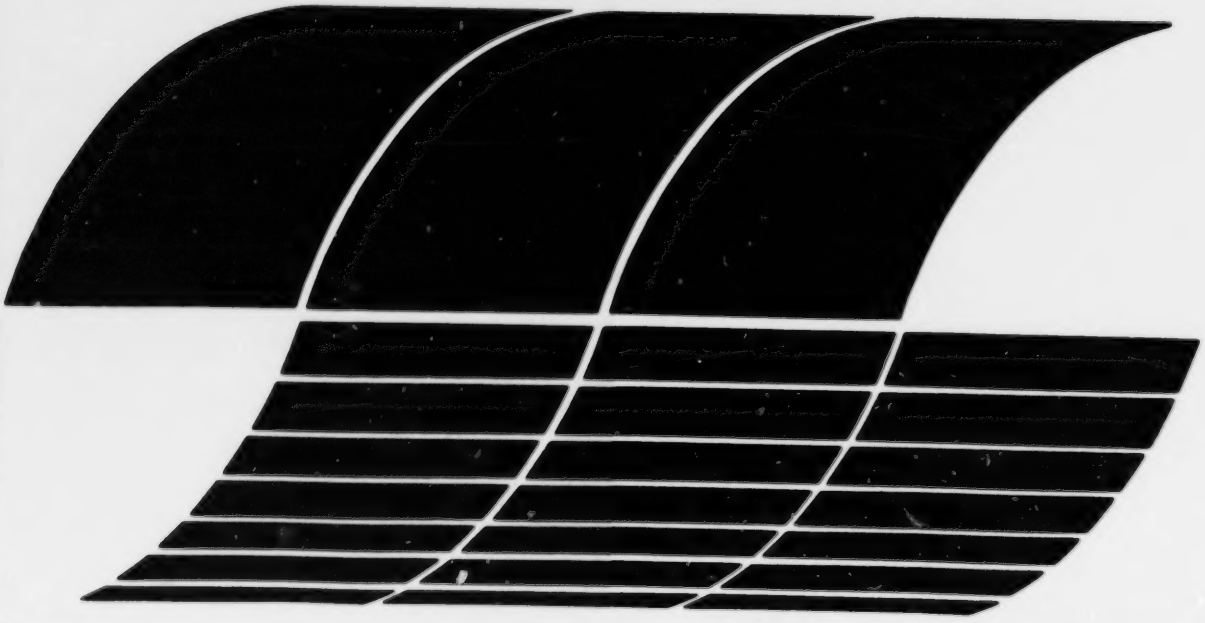
Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

EPA-600/7-80-046a
March 1980

EPA

Coal Gasification/Gas Cleanup Test Facility: Volume I. Description and Operation

Interagency Energy/Environment R&D Program Report



RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into nine series. These nine broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the INTERAGENCY ENERGY-ENVIRONMENT RESEARCH AND DEVELOPMENT series. Reports in this series result from the effort funded under the 17-agency Federal Energy/Environment Research and Development Program. These studies relate to EPA's mission to protect the public health and welfare from adverse effects of pollutants associated with energy systems. The goal of the Program is to assure the rapid development of domestic energy supplies in an environmentally-compatible manner by providing the necessary environmental data and control technology. Investigations include analyses of the transport of energy-related pollutants and their health and ecological effects; assessments of, and development of, control technologies for energy systems; and integrated assessments of a wide range of energy-related environmental issues.

EPA REVIEW NOTICE

This report has been reviewed by the participating Federal Agencies, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Government, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/7-80-046a	2.	3. RECIPIENT'S ACCESSION NO. PB 80 188378
4. TITLE AND SUBTITLE Coal Gasification/Gas Cleanup Test Facility: Volume I. Description and Operation		5. REPORT DATE March 1980 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) J. K. Ferrell, R. M. Felder, R. W. Rousseau, J. C. McCue, R. M. Kelly, and W. E. Willis		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS North Carolina State University Department of Chemical Engineering Raleigh, North Carolina 27650		10. PROGRAM ELEMENT NO. EHE623A 11. CONTRACT/GRANT NO. Grant No. R804811
12. SPONSORING AGENCY NAME AND ADDRESS EPA, Office of Research and Development Industrial Environmental Research Laboratory Research Triangle Park, NC 27711		13. TYPE OF REPORT AND PERIOD COVERED Task Final; 9/77-12/78 14. SPONSORING AGENCY CODE EPA/600/13

15. SUPPLEMENTARY NOTES IERL-RTP project officer is Robert A. McAllister, Mail Drop 61, 919/541-2160.

16. ABSTRACT The report describes an integrated fluidized-bed coal gasification reactor and acid gas removal system. The gasifier operates operates at 100 psig at up to 2000 F, and has a coal feed capacity of 50 lb/hr. The gas cleaning system contains a cyclone, a venturi scrubber, and an absorber/flash-tank/stripper system for acid gas removal. The overall objective of the research carried out using the facility is to characterize completely the gaseous and condensed phase emissions as a function of plant operating conditions. The report contains a detailed description of the plant and associated facilities, a summary of operating procedures, and results of a run for the steam-oxygen gasification of a Western Kentucky No. 11 coal char. By following the outlined operating procedures, the plant can be brought to steady state in less than 4 hours. At steady state, satisfactory material balance closures were achieved on total mass and major elements.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Coal Gasification	Stationary Sources	13H
Gas Scrubbing	Acid Gases	07A
Fluidized Bed Processing		

18. DISTRIBUTION STATEMENT Release to Public	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES
	20. SECURITY CLASS (This page) Unclassified	22. PRICE

EPA Form 2220-1 (9-73)

A7

EPA-600/7-80-046a

March 1980

Coal Gasification/Gas Cleanup Test Facility: Volume I. Description and Operation

by

J.K. Ferrell, R.M. Felder, R.W. Rousseau,
J.C. McCue, R.M. Kelly, and W.E. Willis

North Carolina State University
Department of Chemical Engineering
Raleigh, North Carolina 27650

Grant No. R804811
Program Element No. EHE623A

EPA Project Officer: Robert A. McAllister

Industrial Environmental Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, NC 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

B

CONTENTS

Introduction----------1
Facility Description----------2
Pilot Plant----------2
Gasifier-Particulates Condensables and Solubles (PCS) System Description----------3
Gasifier Start-up and Operation----------16
Acid Gas Removal System (AGRS)----------19
AGRS Start-up and Operation----------32
Alarm and Safety System----------35
Data Acquisition System----------36
Laboratory Facilities and Analytical Program----------39
Laboratory Facilities----------39
Sampling and Sample Preservation----------41
Description of Analytical Methods and Procedures Used----------42
Ash Analysis----------45
Ultimate Analysis----------45
Carbon and Hydrogen Analysis----------45
Sulfur Analysis----------45
Nitrogen Analysis----------46
Gas Analysis----------46
Wastewater Analysis----------47
Ammonia----------47
Nitrogen Analysis----------48
Cyanate Analysis----------48
Cyanide Analysis----------48
Chemical Oxygen Demand Analysis----------48
Thiocyanate Analysis----------48
Phenolics Analysis----------49
Residue Analysis----------49
Sulfide Analsis----------49
Total Carbon, Total Organic Carbon, and Volatile Carbon Analyses----------49
pH Determination----------49
Chloride, Sulfite, Sulfate, and Fluoride Analyses----------50
Sulfur Analysis in Wastewater----------50

Additional Facilities----------51
Coal Research Laboratory----------51
Vapor-Liquid Phase Equilibrium Laboratory----------53
Operation and Results----------54
Gasifier-PCS System----------54
Acid Gas Removal System----------61
References----------68
Appendices----------69
Appendix I----------70
Appendix II----------86
Appendix III----------96
Appendix IV Factors for Unit Conversions----------99

FIGURES

Number		Page
1	Utilities System	4
2	Gasifier-PCS System	5
3	Acid Gas Removal System	6
4	Gasifier Feed Streams	7
5	Gasifier-PCS System	9
6	Fluidized Bed Reactor	11
7	Cyclone Sample Train	14
8	Dehydrators	15
9	Absorber	21
10	Flash Tank	22
11	Stripper	23
12	Start-up Tank, Pumps	24
13	Compressor	25
14	Syngas System	31
15	Data Acquisition and Laboratory Computer System	37
16	Gasifier-PCS Run GO-28	60
17	Column Temperature Profiles and Mass Balances	63
18	AGRS Run AMI-13	65

TABLES

Number		Page
1	Process Control for Gasifier-PCS System	17
2	Process Control for the Acid Gas Removal System	33
3	Analytical Program Summary	43
4	Aqueous Sample Preservation	44
5	Summary of Atomic Absorption Analysis Parameters	52
6	Run GO-28	56
7	Run GO-28	59
8	Run GO-28	62
9	Run A-M-13	67

INTRODUCTION

As a part of a comprehensive long-term program to evaluate the environmental effects of the production of gaseous and liquid fuels from coal, the U. S. Environmental Protection Agency is sponsoring a major research program at North Carolina State University. As a part of the Synthetic Fuels Program, the project is managed by the Industrial Environmental Research Laboratory in Research Triangle Park, North Carolina. The research is carried out by faculty, students and staff of the Chemical Engineering Department of NCSU.

The overall objective of the Project is to characterize completely the gaseous and condensed phase emissions from a coal gasification-gas cleaning process, and to determine how emission rates of various pollutants and process catalyst poisons depend on process parameters. For the early stages of the program, the major emphasis is on the formation and removal of sulfur gases, nitrogen gases and several of the more volatile of the trace metal elements.

In order to satisfy the research objectives in as realistic a manner as possible, the facility used is a small but complete coal gasification-gas cleaning pilot plant. The principal components of the plant are a continuous fluidized bed gasifier; a cyclone separator and a venturi scrubber for removing particulates, condensables, and water-soluble species from the raw synthesis gas; and an absorber, stripper, and flash tank for acid gas removal and solvent regeneration. The gasifier operates at pressures up to 800 kPa (100 psig), has a capacity of 23 kg coal/hr (50 lb/hr), and runs with either steam-air or steam-O_2 feed mixtures. The acid gas removal system is modular in design, so that alternative absorption processes may be

evaluated. Associated with the plant are facilities for direct digital control of all process systems and on-line data acquisition, logging, and graphical display. Facilities for sampling and exhaustive chemical analysis of all solid, liquid, and gaseous feed and effluent streams are also available.

The purpose of this report is to present a detailed description of the facility, a discussion of its operation, and a discussion of the results of a typical run carried out on August 14, 1979, using a Western Kentucky coal char.

FACILITY DESCRIPTION

The facility consists of the pilot plant, several analytical laboratories, and a data acquisition and data reduction computer system. These facilities will be described in the sections that follow.

PILOT PLANT

The pilot plant is designed so that it may be operated as two separate units. The gasifier and particulates, condensables and solubles (PCS) removal system may be operated as a unit with the gas produced going directly to a gas disposal flare. The acid gas removal system (AGRS) may be operated as a unit using a feed gas made up by mixing various gases in a gas mixing manifold. The entire system may be also operated as an integrated unit with a portion of the gas produced in the gasifier used as feed gas to the AGRS. In all cases, gaseous effluent streams are combined in a vent gas header and sent to the flare for disposal.

Simplified, schematic diagrams of the plant are shown in Figures 1

through 3. Figure 1 shows a diagram of the gas feed utilities system which is used to provide preheated gas to the gasifier and also to provide a mixture of gases to the AGRS when it is operated as a separate unit. Figures 2 and 3 are diagrams of the gasifier-PCS system and the AGRS, respectively.

The remainder of this section will be devoted to a detailed description of the pilot plant facility.

Gasifier-PCS System Description

A detailed schematic diagram of the utilities system, which provides preheated gases to the gasifier is shown in Figure 4. Saturated steam at 150 psig is available from the University Power Plant. The steam flow rate is indicated by FT-115, using an orifice meter, and is controlled by FCV-115. The steam is superheated in an electrically heated superheater, H-14, to approximately 1,000°F. Manual valves, V-140 and V-141 provide a means for bypassing the steam to a condenser during gasifier start-up and for calbration of FT-115.

Oxygen flow (or air flow) is indicated by FT-114, using a laminar flow element, and is controlled by FCV-114. Nitrogen flow is indicated by FT-116, using a laminar flow element, and is controlled by FCV-116. The N_2 and O_2 (or air) streams combine and are preheated to approximately 1,000°F in an electrically heated preheater, H-13. The superheated steam and the preheated gases combine before being fed to the gasifier.

The maximum oxygen consumption during gasifier operation is less than 5 SCFM (32°F and 1 atm.) and a manifold of six cylinders provides an adequate supply. Nitrogen for the entire plant is supplied by a 6,000-gallon liquid nitrogen tank and vaporizer system.

All three flow meters are calibrated in place and at pressures very

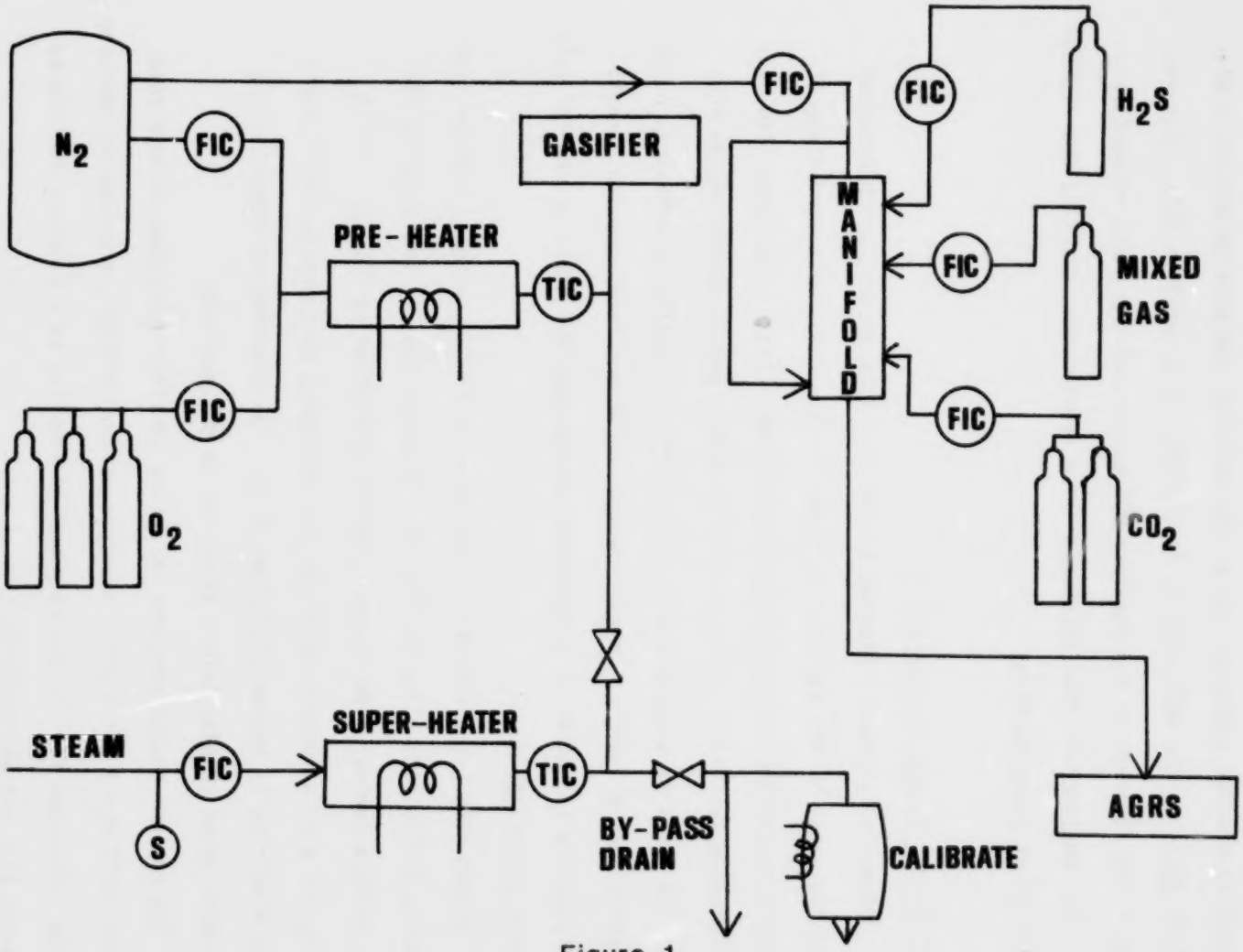


Figure 1
Utilities System

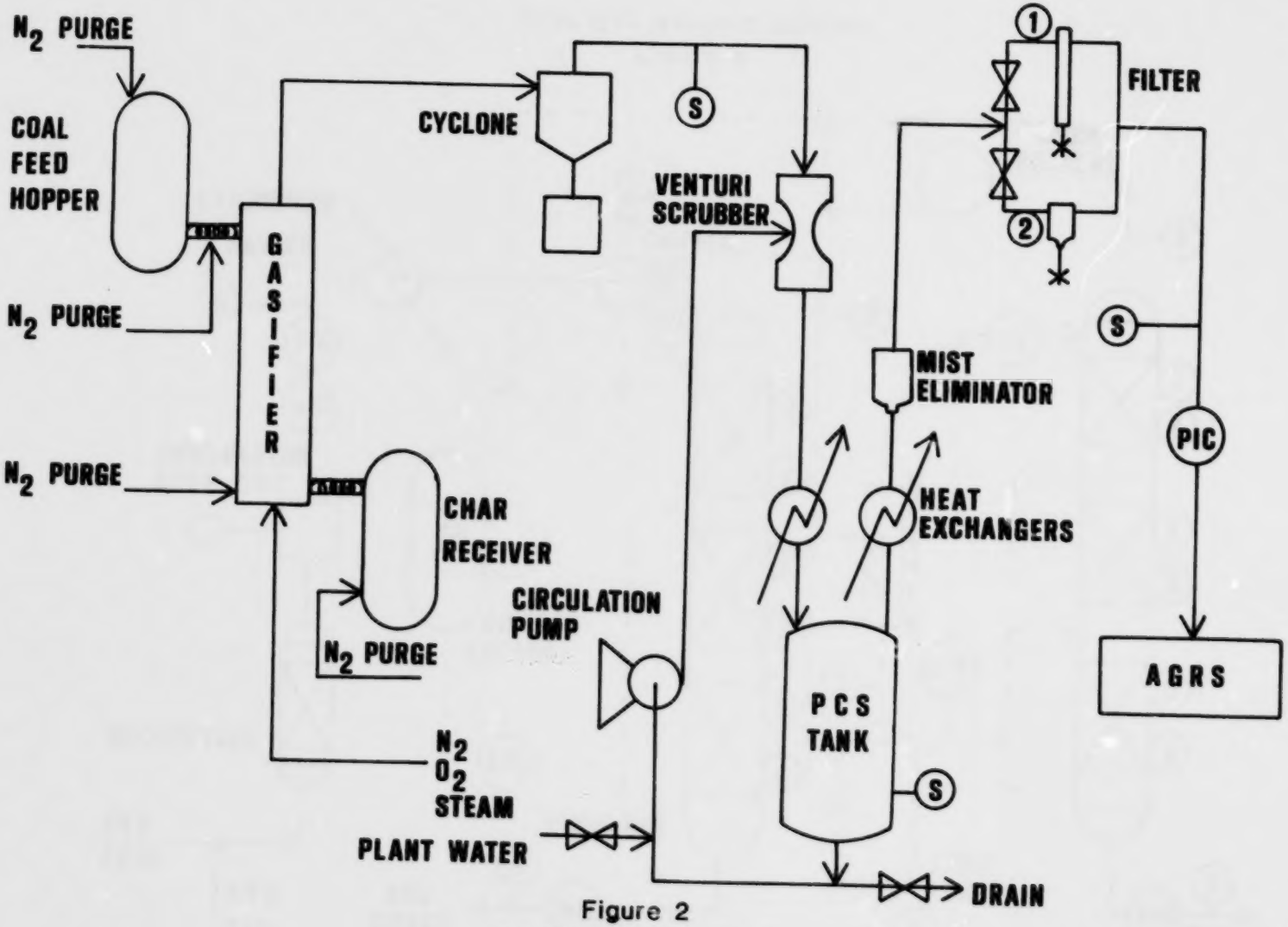


Figure 2
Gasifier-PCS System

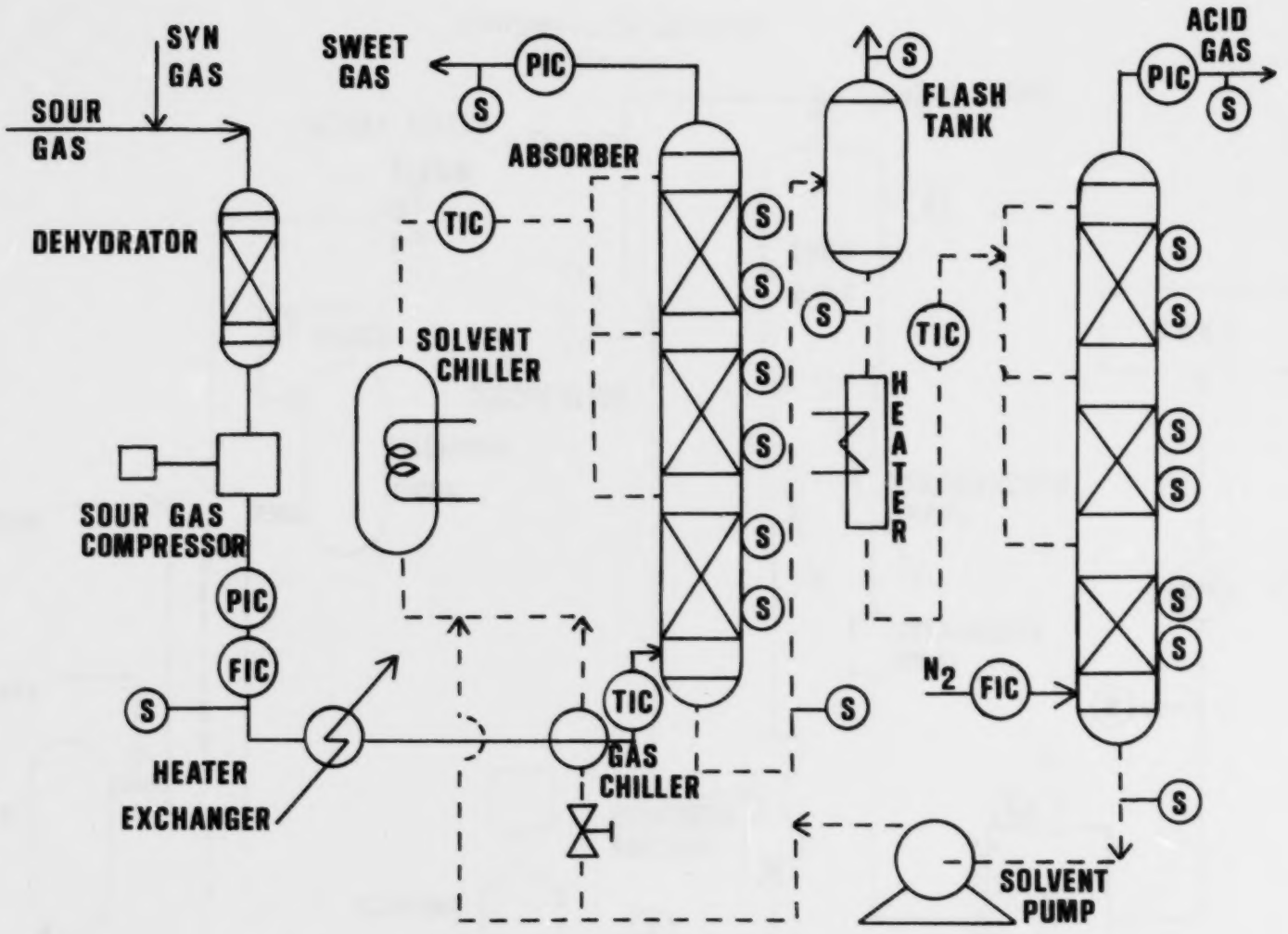


Figure 3
Acid Gas Removal System

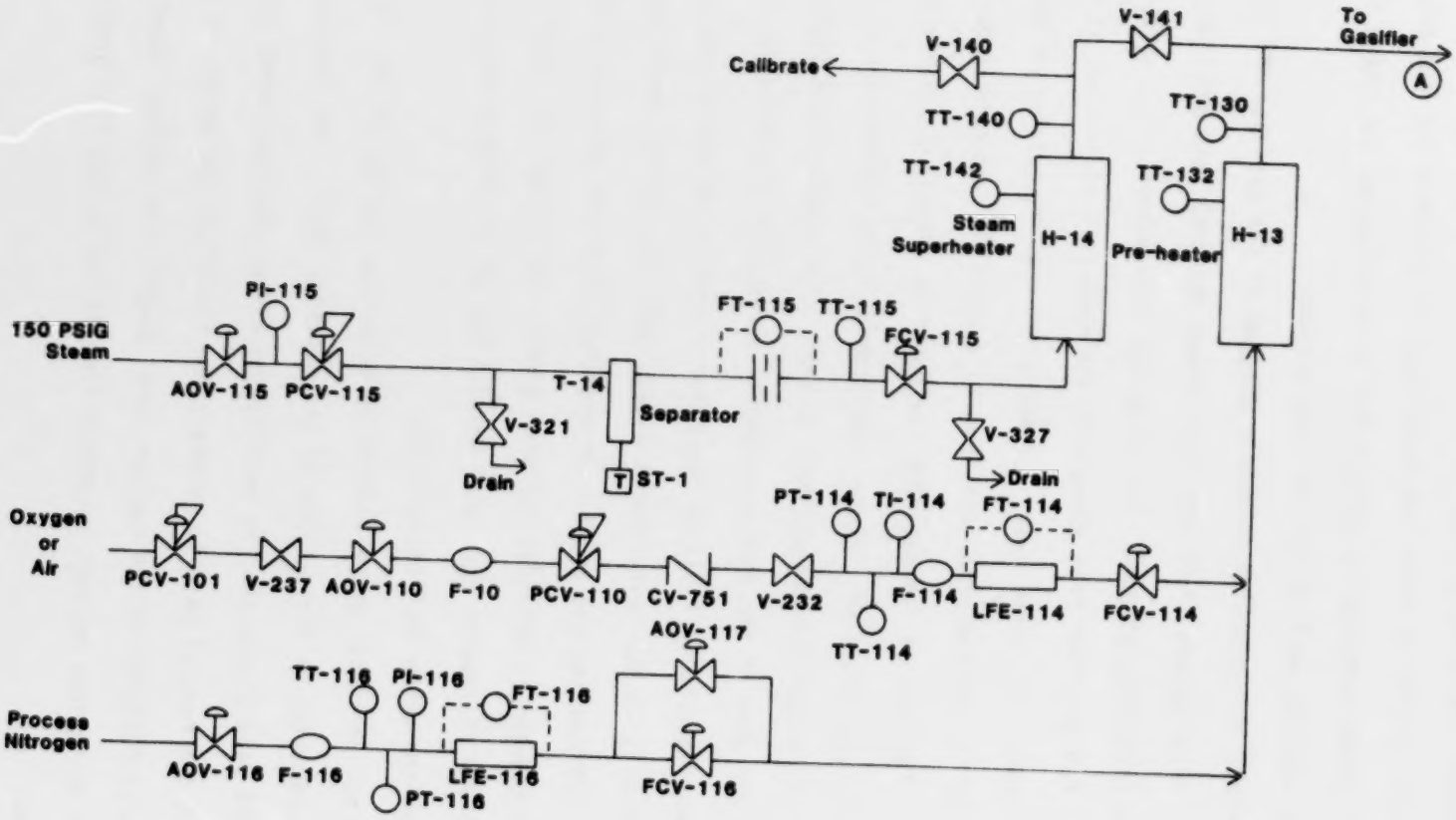


Figure 4

Gasifier Feed Streams

near to normal operating pressures. In addition, temperature and pressure transmitters located at each flow meter make it possible for the data acquisition system computer to correct the calibration and compute a true mass flow rate for each of the gas feed streams.

Figure 5 is a detailed schematic diagram of the gasifier-PCS system. Hydraulic drive motors, P-01 and P-02, power the coal feed and char extractor screw conveying systems. Coal from the feed hopper, T-01, enters the fluidized bed gasifier from above with the feed rate controlled at the main control panel. The screw used in feeding the coal is calibrated and the calibration is checked after each run to account for changing capacity due to wear. The char is removed from the gasifier through a similar system into char receiver, T-02. Again, the removal screw calibration is frequently checked to account for changes due to wear. The speed of the hydraulic motors, and thus of the feed and removal screws, is controlled by regulating the flow of hydraulic fluid to the motor. Both screw speeds are controlled automatically at the main control panel, with the desired revolutions per minute (RPM) as the set point. In addition to an RPM display, a total revolution counter is provided for each screw. For a run, the total weight of coal fed divided by the total revolutions of the feed screw provides a check on the screw feed calibration.

A nuclear level gauge capable of detecting the top of the fluidized bed, is mounted on the outside of the gasifier shell. The operator sets the feed rate of the coal by setting the RPM of the feed screw and then adjusts the removal rate to maintain the fluidized bed height in a 2-inch window as measured by the nuclear level gauge. The nuclear level gauge can be set at various heights to permit flexibility in gasifier operating conditions.

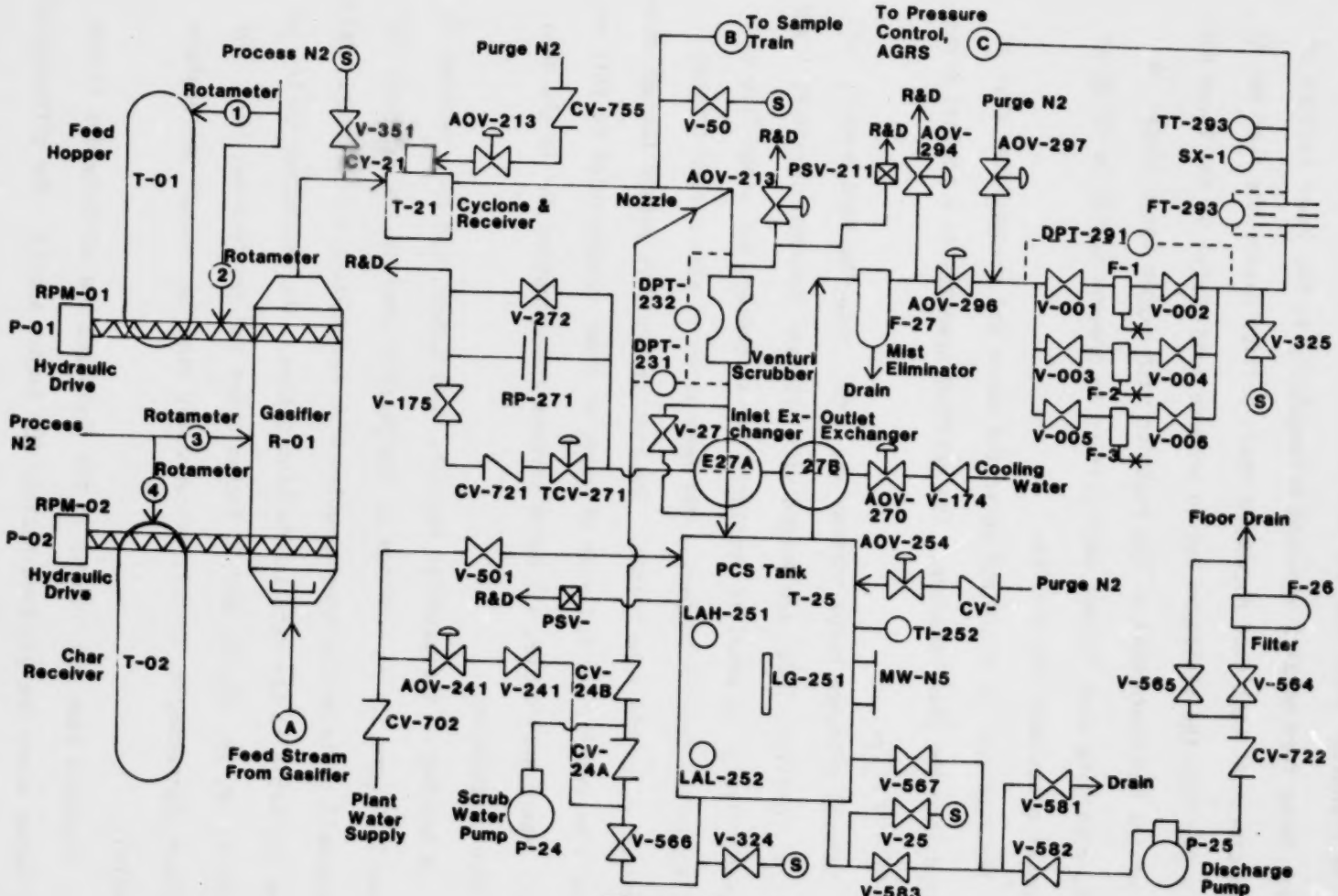


Figure 5

Gasifier-PCS System

The preheated gases and the superheated steam are fed to the gasifier through three feed nozzles arranged triangularly in the lower section of the reactor. At each feed nozzle, a small orifice causes the feed gas to form a jet into the fluidized bed to promote good mixing in the lower portion of the bed where most of the reaction with oxygen takes place. A short, diverging cone follows each orifice to prevent solids build-up on the metal parts near the orifice.

The feed coal is fluidized above the cones and the gasifier char is removed into the char receiver. The gasifier operates at a nominal pressure of 100 psig.

Several nitrogen purge streams are used. Both the feed hopper, T-01, and char receiver, T-02, are purged with nitrogen through rotameters 1 and 4, respectively, to equalize pressures and to prevent any steam from condensing inside the two hoppers. The feed screw is also purged through rotameter 2 for the same reason. The reactor shell is purged through rotameter 3 to prevent any build-up of water or other condensables in this section. Where applicable, all purge streams are accounted for in the mass balance calculation.

A drawing of the gasifier reactor, feed hopper, and char receiver is shown in Figure 6. In addition to the details shown on the drawing, the thermowell contains six thermocouples with temperature transmitters, making them available to the data acquisition system. The thermocouples are located 5, 10, 25, 35, 45, and 55 inches above the gas feed nozzles. The 10-inch thermocouple, TT-201, is currently used for reactor temperature control.

Pressure taps are located in the fluidized bed at distances 15 and 35 inches above the gas feed nozzles (20 inches apart). The differential

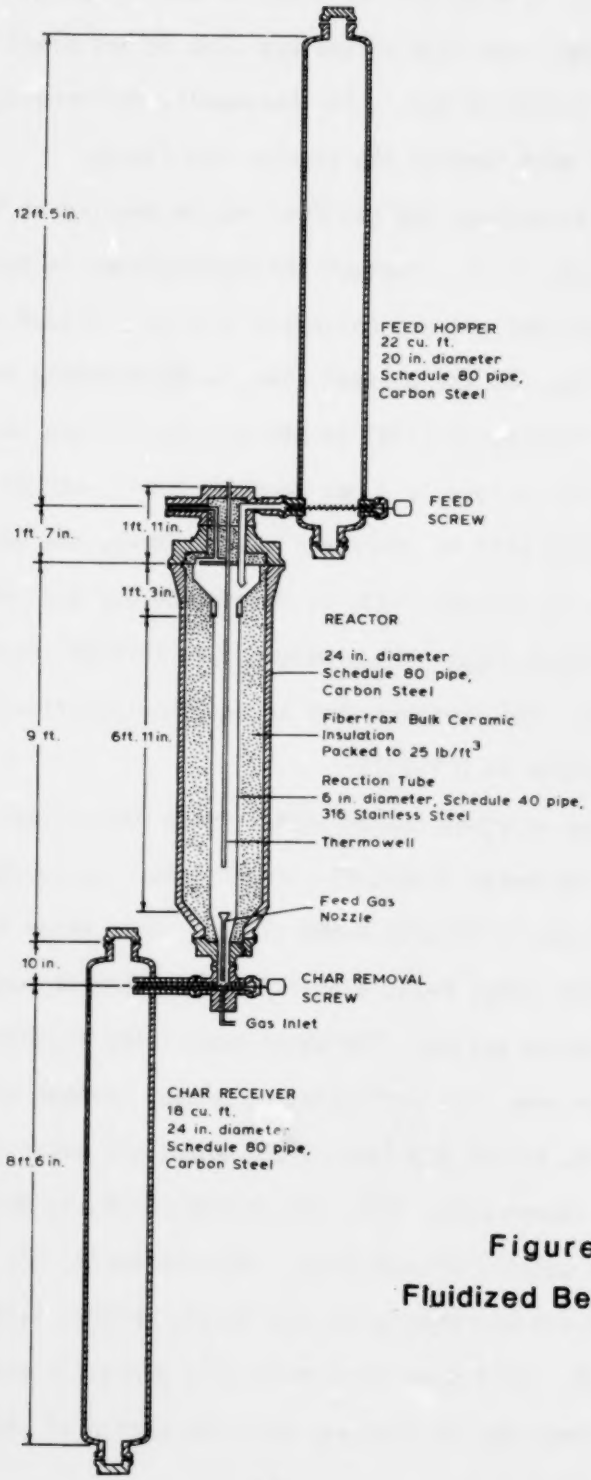


Figure 6

Fluidized Bed Reactor

pressure, DPT-201, is also available at the control panel and to the data acquisition system. Gas sample taps may also be installed at any location in or above the fluidized bed. The thermowell, differential pressure, and gas sample lines exit through the reactor top flange.

The raw gases exiting the gasifier may be sampled at V-351 before entering the cyclone, CY-21. Most of the particulates in the gas stream are removed in the cyclone and are collected in a pressurized receiver, T-31, for later sampling. At the present time, a thermocouple and temperature transmitter are located at V-351 to monitor the cyclone temperature.

Following the cyclone is a gas sampling train, marked B on Figure 5. This sampling train will be described later. Next, the gas passes through a venturi scrubbing system, S-23, to quench the gas and remove most of the solubles and condensables. The pressure drop through the scrubber is measured at DPT-232. The pressure drop across the injection nozzle to the scrubber is measured at DPT-231.

Water to the scrubber can be either fresh water from the building supply or recycled water from T-25. Fresh water can be fed to the nozzle through AOV-241 and V-241 with V-566 closed. This water is then pumped by the scrub water pump, P-24, a positive displacement pump, to the nozzle of the scrubbing system. The water containing solubles and condensables (along with some fine particulates) passes through the inlet heat exchanger, E-27A, to the PCS tank, T-25, where the water is later drained through the discharge pump, P-25, and filter, F-26, to disposal. This material can be sampled through V-25. Alternatively, the material drained to the PCS tank can be recycled to the nozzle through V-566 at the scrub water pump, P-24. This mode is particularly useful in watching the build-up of various compounds of interest over the period of the run. The re-

cycle stream can be sampled at V-324.

The sour gas leaving the PCS tank is further cooled in outlet heat exchanger, E-27B, to remove any residual condensables. A mist eliminator, F-27, and a series of filters, F-1, 2, 3, are used to insure that all fine particles are removed before leaving the PCS system. Two coalescing filters, F-1 and F-2, are used with only one filter being in-line at any time. As the pressure drop across the filter increases, DPT-291, the other filter can be switched in-line while the plugged filter is being reconditioned.

The sour gases can be sampled manually at V-325 before being sent to the Acid Gas Removal System. The on-line analytical equipment is used to monitor oxygen level and gas composition by a small, continuous gas stream at SX-1. The sour gas flow rate is measured by an orifice meter and flow transmitter, FT-293.

A gas sampling train is located just downstream of the cyclone; a schematic diagram is shown in Figure 7. The sampling train continuously withdraws a small gas stream (25 SCFH) from the cyclone exit through a condenser and filter to provide a cool, dried gas stream and a sample of condensate. After the gas has been conditioned, both high and low pressure gas sample ports are available. The sample train also provides a gravimetric measure of the water in the gasifier effluent by measuring the dry gas flow rate with a test meter, and the condensate flow rate.

Pressure control for the gasifier-PCS system is accomplished by controlling the flow of sour gas at PCV-310, shown on Figure 8. Since the gas volume of the gasifier-PCS system is approximately 90 cubic feet and the pressure sensor, PT-310, is located near the control valve, good pressure control usually means that the sour gas flow rate, as measured by FT-

Figure 7

Cyclone Sample Train

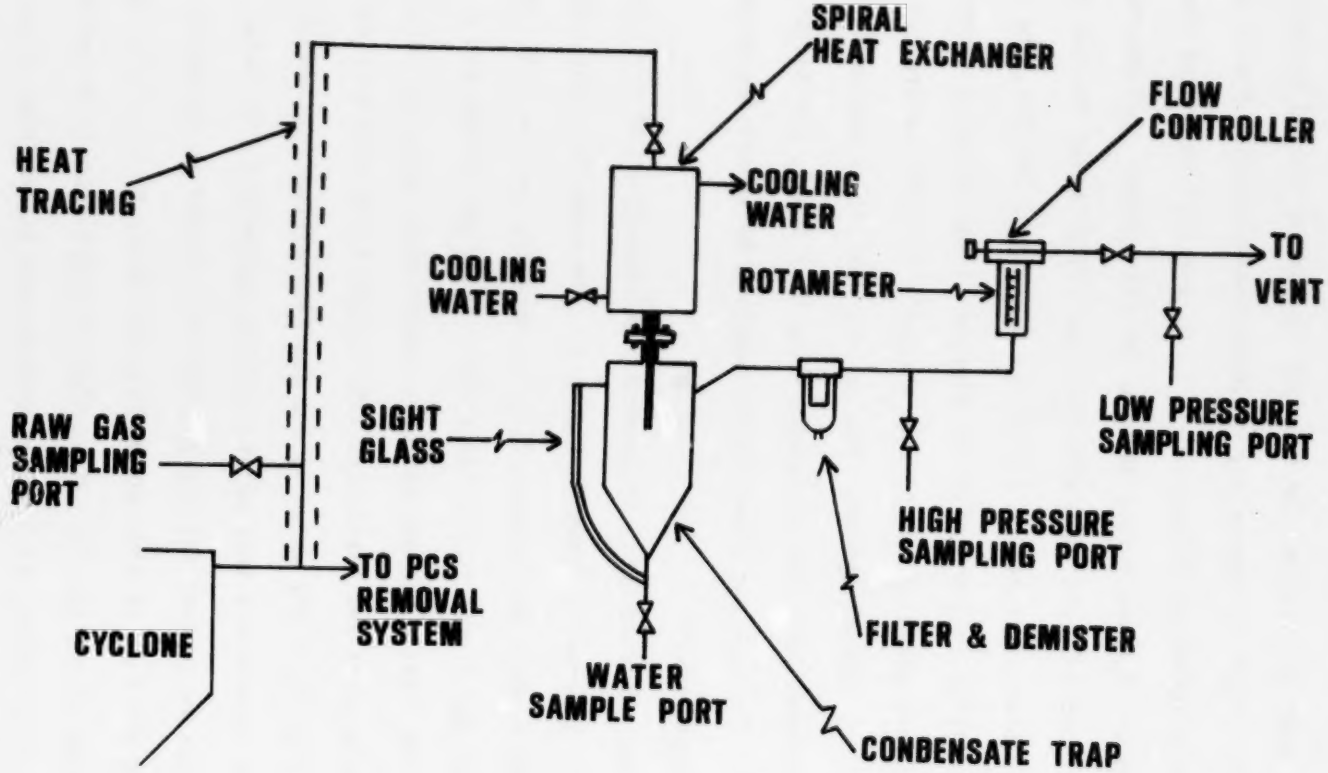
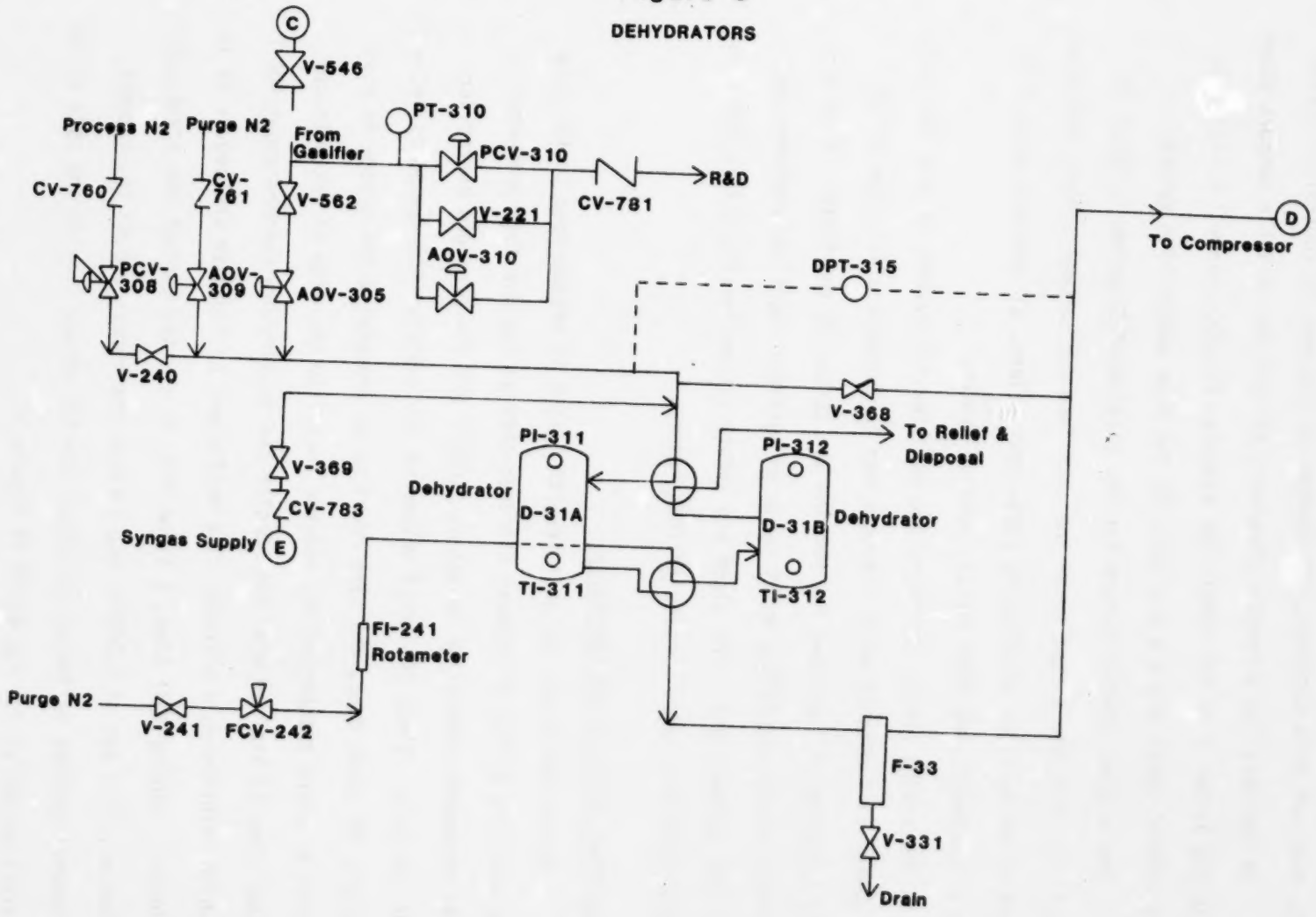


Figure 8

DEHYDRATORS



293, does not hold constant, but changes in response to controller action.

On the detailed schematic diagrams, all process variable sensors shown with the letter T in the label, for example, PT-310, transmit a signal to the control panel and are available to the data acquisition system.

The process control scheme for the gasifier-PCS system is shown in Table I. With the exception of the coal feed and removal screws, which are controlled by independent solid state controllers, all process control is by a Honeywell, TDC-2000 digital control system.

The entire plant, including the gasifier-PCS system, is provided with a number of locations where nitrogen may be introduced into the system and a number of locations for venting the system to the flare. There are several alarm conditions which cause an automatic vent and nitrogen purge of the entire plant. The alarm and safety system for the entire plant is described in a later section of this report.

Gasifier Start-up and Operation

During start-up, the gasifier-PCS system is pressurized to 100 psig by starting a flow of process nitrogen through the gas feed preheater. The preheater controller is set at 1,000°F and the pressure controller at 100 psig. Since the total volume of the gasifier-PCS system is approximately 90 cubic feet, the time required to pressurize the system is also used to start preheating the reactor vessel by the flow of hot nitrogen. Coal feed is started when the reactor bed temperature reaches about 400°F while maintaining a nitrogen flow sufficient to fluidize the bed as it is formed. During this time, a steam flow is started through the steam superheater, also set at 1,000°F, and through the bypass around the reactor. Several process variables are shown plotted versus time during part of the start-up period for run GO-28 in Figure 16.

Table I. Process Control for Gasifier-PCS System

Control Variable	Sensor Transmitter	Control Element	Controller
Steam Flow	FT-115	FCV-115	TDC-2000
Oxygen Flow	FT-114	FCV-114	TDC-2000
Nitrogen Flow	FT-116	FCV-116	TDC-2000
Steam, Inlet Temperature	TT-140	Heater Element H-14	TDC-2000
Gas Inlet Temperature	TT-130	Heater Element H-14	TDC-2000
Gasifier Temperature	TT-201	Cascade to O_2 Flow Control	TDC-2000
Gasifier Pressure	PT-310	FCV-310	TD-2000
Coal Feed Screw	RPM-01	Hydraulic Fluid Flow Control Valve	Barber-Colman 520 Solid State Controller
Char Removal Screw	RPM-02	Hydraulic Fluid Flow Control Valve	Berber-Colman 520 Solid State Controller

When the bed temperature has reached approximately 700°F, and with the bed height between 20 and 30 inches, a small flow of oxygen is started. At this temperature, the bed will almost always ignite and after ignition, the bed temperature is brought to approximately 1,450°F by slowly increasing oxygen flow. At this temperature, steam flow is introduced by switching an established small flow of superheated steam at 1,000°F into the reactor by closing V-140, allowing the steam pressure in the superheater to build up to 110 psig, and opening V-141. To achieve the desired steady state conditions, nitrogen flow is gradually decreased,

steam flow is increased, and oxygen flow is adjusted to maintain the reactor temperature at the desired value. All of the above changes must be done smoothly, and good results have been achieved by making adjustments so that only gradual changes in the calculated superficial gas velocity, and reactor temperatures occur.

The steady state coal feed rate is established by controlling the speed of the coal feed screw to maintain the desired feed rate, and adjusting the removal screw speed to maintain the desired bed height as indicated by the bed level gauge. During the start-up phase, the bed height can be followed very well by the temperature sensors located in the bed, by the bed differential pressure measurement, and by the nuclear level gauge. During every change made during start-up, all of the variables, with the exception of the reactor temperature, are in automatic control and changes are made by changing set points. When near steady state conditions have been achieved, the reactor temperature is put in automatic control by cascading the temperature control loop to the set point of the oxygen flow control loop. Experience with this method of reactor temperature control has been very good.

When the bed is well fluidized, the process described above works very well and reactor start-up is fast and smooth. For a variety of reasons, the bed is often not well fluidized during the start-up period and a variety of difficulties can occur. The most probable causes and effects are hot spots due to poor mixing in the bed, a dense bed which may be lifted to the top of the reactor, and probably, many others. It should be noted that where a good steady state is obtained, the operation is very stable and cannot be easily upset. A possible reason for the difficulty of operation dur-

ing start-up, and ease of operation during steady state, is the difference in the manner in which the fluidizing gas velocity is generated. During start-up, with no reaction in the bed, all of the fluidizing gas emerges from the three feed nozzles and may not be well distributed. During operation, the carbon-steam reaction results in an increase in the volume flow of gas and the carbon-oxygen reaction increases the gas temperature in a zone just above the cones. Both of these factors act to provide a fluidizing gas velocity within the bed and with good distribution across the bed. Both the start-up and the steady state operations of one run, GO-28, will be discussed later in this report.

Complete startup and operating procedures and checklists, are given in Appendix I in outline form.

Acid Gas Removal System

The major components of the Acid Gas Removal System (AGRS) are an absorbing column, a flash tank and a stripping column, with the accompanying auxiliary equipment to permit operation using a number of different absorbing solvent systems. The system has sufficient flexibility to operate using at least four solvents, methyl alcohol, dimethylether of polyethyleneglycol (DMPEG), hot potasium carbonate, and monoethanolamine. Detailed schematic diagrams of the AGRS are given in Figures 8 through 14.

The system will be described by first following through the liquid solvent circuit, and then following the gas streams.

Regenerated solvent enters the absorber, C-34, at one of three points, depending upon the number of sections of packing to be used in a given set of experiments. The solvent inlet temperature is measured at TT-349A. The solvent flows down the column contacting the gas flow in either one,

two, or three 7-foot sections of 1/4" ceramic Intalox saddles. Two solvent sample ports are located within each section of packing; sample points are located to reduce end effects. The temperature profile in the column is available from two temperature transmitters in each packed section. The pressure drop across the column packing is monitored by DPT-340 and column pressure is measured at PT-345.

The acid gas-rich solvent drains into an absorber bottoms reservoir where the liquid level, LT-341, is controlled by LCV-341 located at the flash tank entrance. Before entering the flash tank, T-35, the solvent can be routed through a filter, F-31, if necessary. The solvent can be sampled manually prior to the filter (or the filter bypass). Pressure drop around the filter is measured at DPT-310. After passing through the filter (or bypassing the filter through V-526), the solvent enters the flash tank through LCV-341. Flash tank pressure is measured at PT-353. Level in the flash tank is regulated by LCV-351, at the stripper inlet, which receives its signal from LT-351 at the flash tank. It is also possible to by-pass the flash tank using valve V-529. When operated in this manner the liquid level in the absorber is regulated using LCV-351 at the stripper. The solvent leaving the flash tank can be sampled manually and its temperature is measured by TT-375.

The solvent leaving the flash tank will take one of a number of routes depending upon the solvent system being used:

(1) Methanol: The solvent leaving the flash tank is passed through the Lean/Rich exchanger, E-37, before entering the stripper, C-39. Here, the rich methanol exiting the flash tank exchanges heat with the lean methanol leaving the stripper. This warms the inlet methanol to the stripper for better

Figure 9

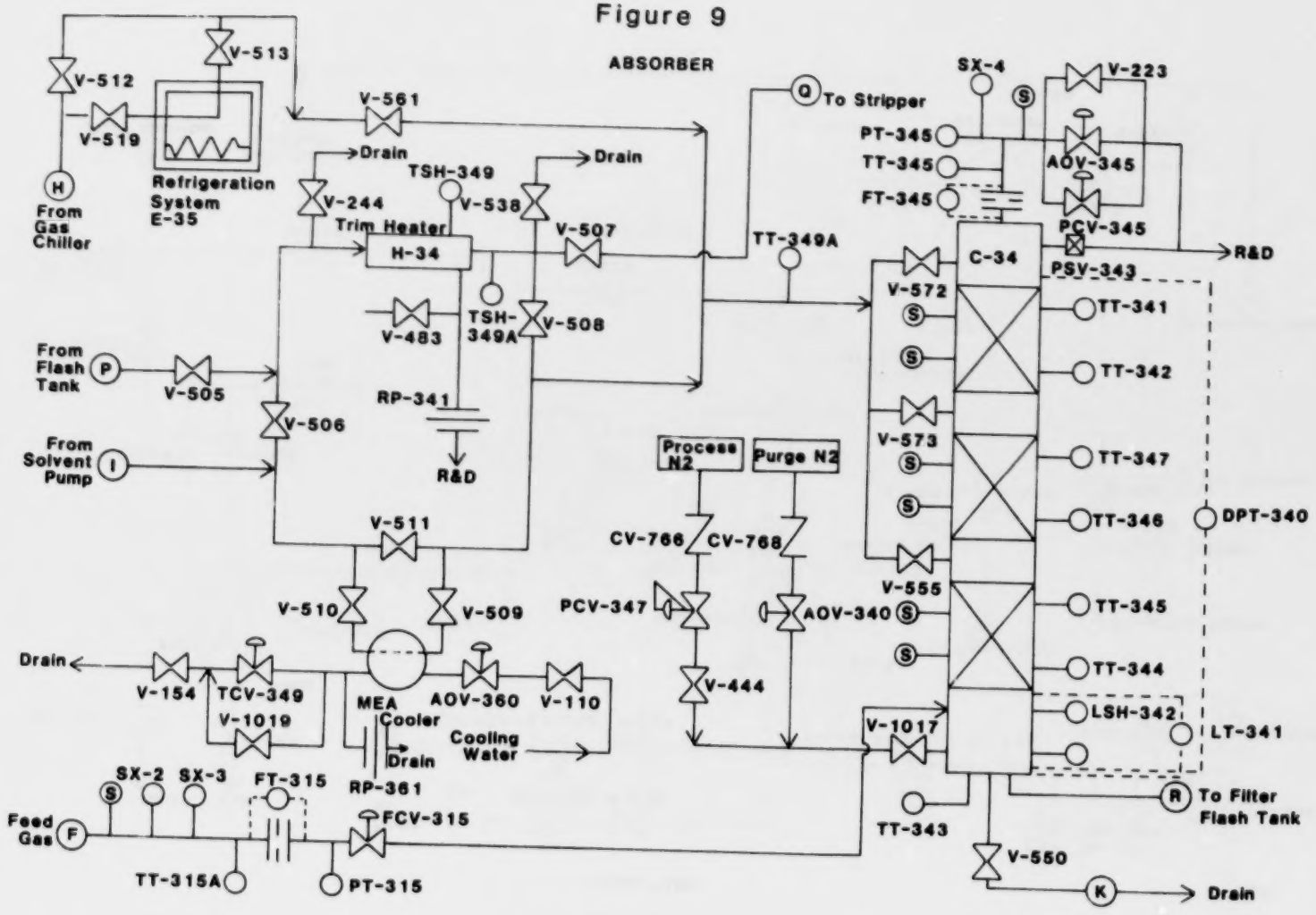

ABSORBER
V-513
V-512
V-519
V-561
Refrigeration System E-35
From Gas Chiller
Drain
TSH-349
Drain
V-244
Trim Heater
H-34
V-538
V-507
TT-349A
To Stripper
SX-4
V-223
PT-345
TT-345
FT-345
AOV-345
PCV-345
R&D
C-34
PSV-343
V-572
TT-341
TT-342
V-573
TT-347
TT-346
DPT-340
V-555
TT-345
TT-344
LSH-342
LT-341
V-1017
To Filter Flash Tank
TT-343
V-550
Drain
V-483
TSH-349A
V-508
From Flash Tank
V-505
RP-341
V-506
From Solvent Pump
R&D
Process N2
Purge N2
CV-766
CV-768
PCV-347
AOV-340
V-444
V-511
V-510
V-509
Drain
V-154
TCV-349
V-1019
MEA Cooler
AOV-360
V-110
Drain
Cooling Water
RP-361
SX-2
SX-3
FT-315
Feed Gas
FCV-315
TT-315A
PT-315

Figure 10

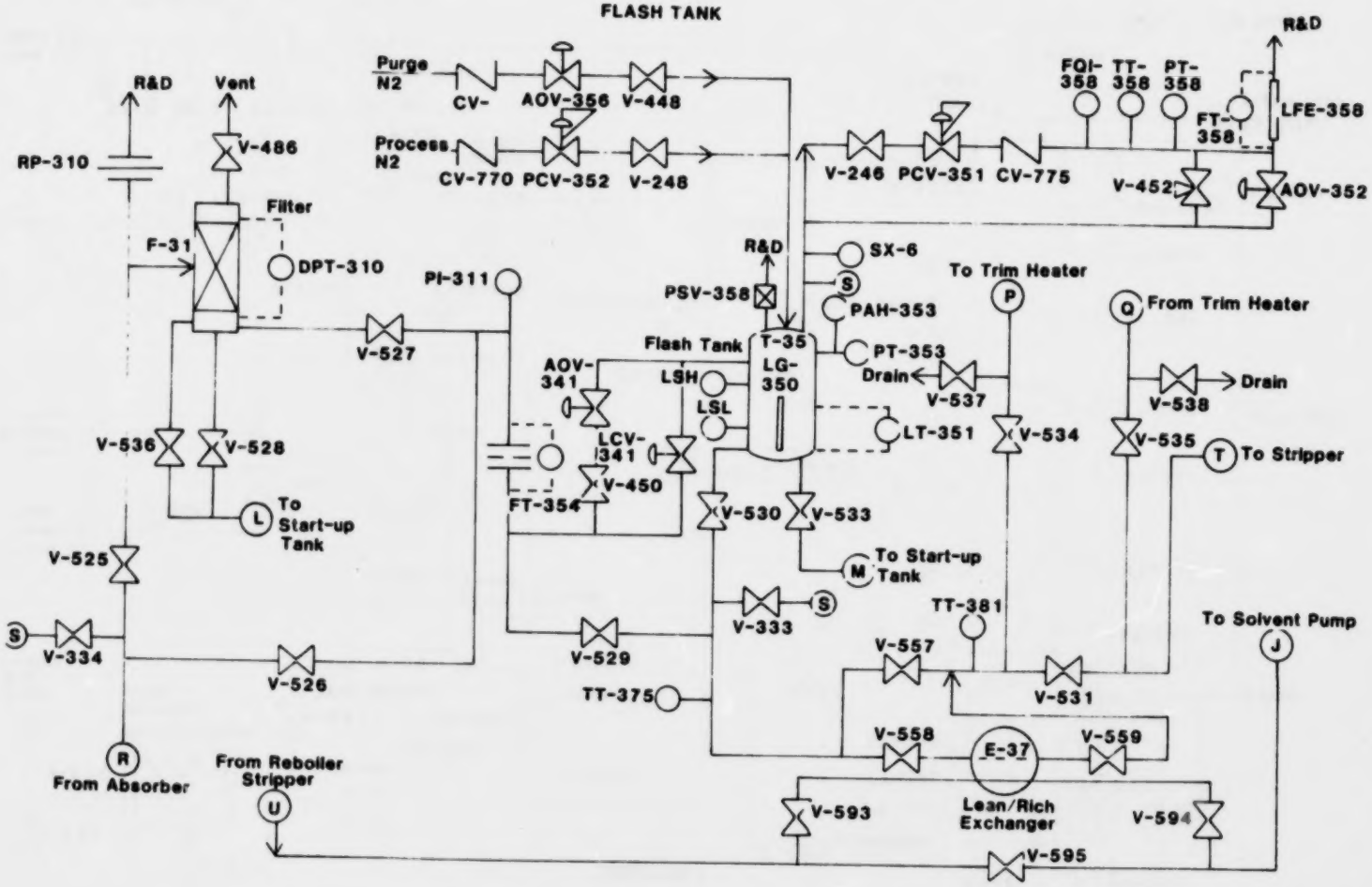
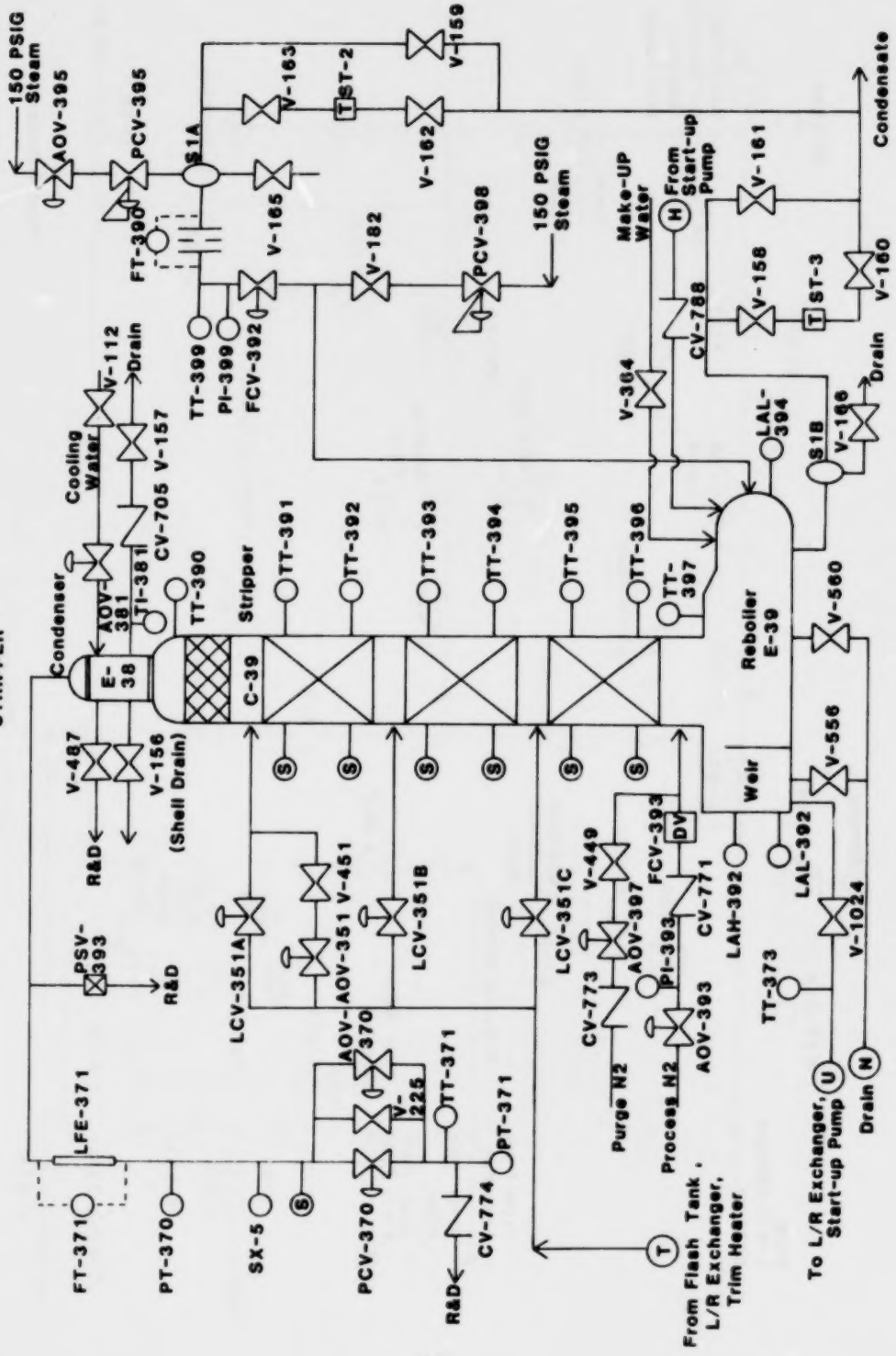

FLASH TANK
R&D
Vent
Purge N2
CV-
AOV-356
V-448
Process N2
CV-770
PCV-352
V-248
FQI-358
TT-358
PT-358
FT-358
LFE-358
V-246
PCV-351
CV-775
V-452
AOV-352
RP-310
V-486
Filter
F-31
DPT-310
PI-311
V-527
R&D
SX-6
PSV-358
S
PAH-353
To Trim Heater
P
Q
From Trim Heater
Flash Tank
T-35
LG-350
PT-353
Drain
V-537
V-538
Drain
AOV-341
LSH
LSL
LT-351
V-534
V-535
T
To Stripper
LCV-341
V-450
FT-354
V-536
V-528
L
To Start-up Tank
V-530
V-533
M
To Start-up Tank
V-525
S
V-334
V-526
V-529
V-333
S
TT-381
To Solvent Pump
J
V-557
V-531
TT-375
V-558
E-37
V-559
R
From Absorber
From Reboiler Stripper
U
V-593
Lean/Rich Exchanger
V-594
V-595

Figure 11


STRIPPER
150 PSIG Steam
AOV-395
PCV-395
S1A
V-163
ST-2
V-159
V-162
150 PSIG Steam
PCV-398
V-182
V-165
FT-390
TT-399
PI-399
FCV-392
V-112
Drain
Cooling Water
V-157
CV-705
AOV-381
TI-381
TT-390
Condenser
E-38
Stripper
C-39
TT-391
TT-392
TT-393
TT-394
TT-395
TT-396
TT-397
Reboiler E-39
V-560
V-487
V-156 (Shell Drain)
R&D
PSV-393
LFE-371
FT-371
PT-370
SX-5
LCV-351A
AOV-351
V-451
LCV-351B
AOV-370
V-225
TT-371
PT-371
PCV-370
CV-774
LCV-351C
V-449
AOV-397
CV-773
Purge N2
Process N2
AOV-393
FCV-393
DV
CV-771
PI-393
LAH-392
LAL-392
Weir
V-556
TT-373
V-1024
From Flash Tank, L/R Exchanger, Trim Heater
To L/R Exchanger, Start-up Pump
Drain
Make-UP Water
From Start-up Pump
V-161
Condensate
V-158
ST-3
V-160
CV-788
V-364
LAL-394
S1B
V-166
Drain

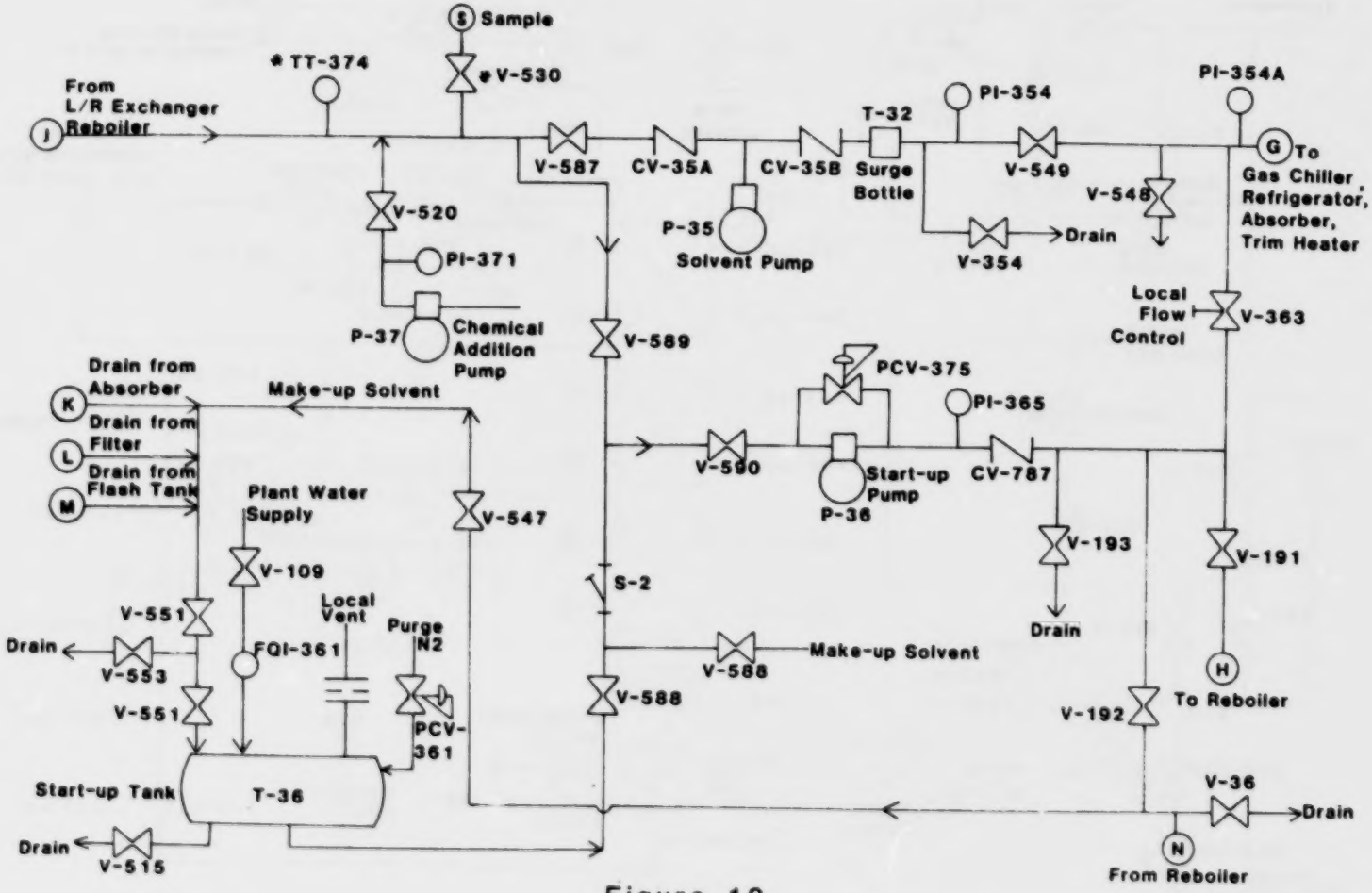
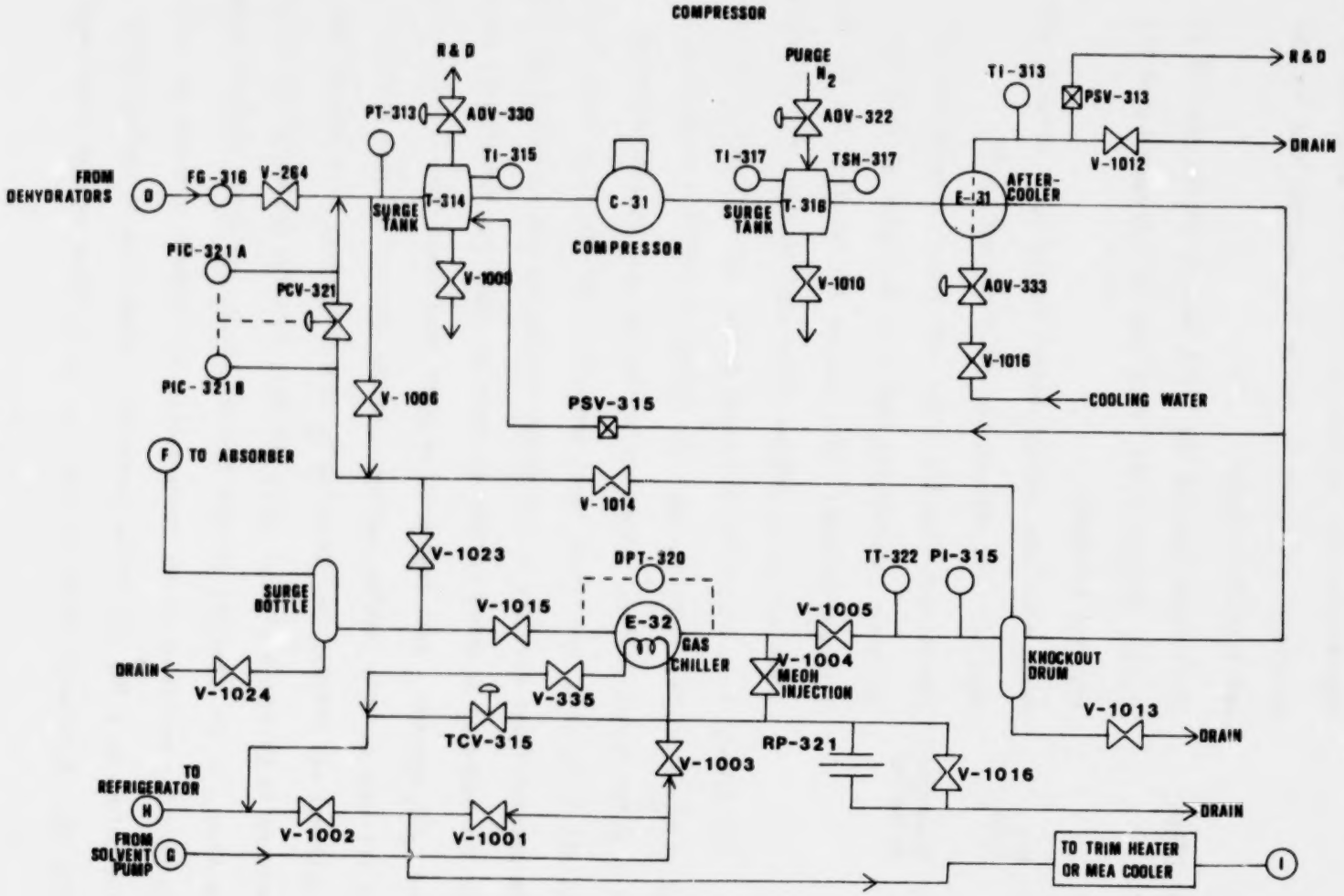


Figure 12

Start-Up Tank, Pumps

Figure 13


COMPRESSOR
R & D
PURGE
N_2
PT-313
AOV-330
AOV-322
TI-313
PSV-313
R & D
DRAIN
V-1012
TI-315
TI-317
TSH-317
FROM DEHYDRATORS
D
FG-316
V-264
SURGE TANK
T-314
C-31
COMPRESSOR
SURGE TANK
T-318
E-31
AFTER-COOLER
PIC-321A
PCV-321
V-1009
V-1010
AOV-333
V-1016
COOLING WATER
PIC-321B
V-1006
PSV-315
F
TO ABSORBER
V-1014
V-1023
DPT-320
TT-322
PI-315
SURGE BOTTLE
V-1015
E-32
GAS CHILLER
V-1005
V-1004
MEOH INJECTION
KNOCKOUT DRUM
DRAIN
V-1024
V-335
TCV-315
V-1013
DRAIN
RP-321
V-1003
V-1016
TO REFRIGERATOR
H
DRAIN
V-1002
V-1001
FROM SOLVENT PUMP
G
TO TRIM HEATER OR MEA COOLER
I

regeneration and, at the same time, takes advantage of the desorption endotherm to reduce the cooling load for the refrigeration system.

(2) DMPEG: The solvent leaving the flash tank by-passes the L/R exchanger through V-557, V-531 and is introduced directly into the stripper.

(3) Hot K_2CO_3: Here, also, the solvent enters the stripper directly and does not pass through the L/R exchanger, E-37.

(4) Monoethanolamine: The solvent from the flash tank passes through the L/R exchanger, exchanging heat with the material from the stripper bottoms. This reduces the heating load for the steam stripping process while cooling the stripper bottoms.

The solvent from any of the processes enters the stripper, C-39, at one of three points, depending upon the number of sections of packing to be used for mass transfer. Flow rate to the stripper is controlled by LCV-351 A, B, or C, the liquid level controller for the flash tank, T-35. The solvent flows down the column either contacting the stripping gas flow for the methanol or DMPEG systems or reboiled vapor for monoethanol amine and K_2CO_3 systems. Both regenerative modes take place over 7-foot sections of 1/4-inch ceramic Intalox saddles. In the physical solvent systems (methanol and DMPEG), the stripping N_2 flow originates from AOV-393 and is metered in through a digital valve, DV-393, into the bottom of the column packing. In the chemical solvent systems (K_2CO_3 and MEA), 150-psig steam is used for reboiling, E-39; condensables are removed from the gas exiting the column by E-38, the reflux condenser. Steam flow is controlled by FCV-392. Additional steam for start-up can be added through V-182 regulated

at PCV-398. The acid gas then passes to the flare through the appropriate monitoring and sampling ports. The column is operated at total reflux.

The lean solvent leaving the stripper bottom will take different routes depending upon the solvent system used. P-35, with a capacity of 2 GPM, can deliver the solvent at pressures in excess of 500 psig to the pressurized absorber, C-34. It is a positive displacement pump with the pumping action derived from the opening and closing of check valves 35-A and 35-B. A surge bottle is required downstream of the pump discharge, T-32, to damp pulsations arising from pump operation. The lean solvent fed to the pump can be sampled manually at V-530. A chemical addition pump, P-37, can be used to meter in surfactants or other additives up-stream of the solvent circulation pump at V-520.

To reduce the cooldown time for the physical solvent systems (methanol and DMPEG), a start-up pump is also used. This is a Viking gear pump with a capacity of 5.6 GPM which makes better use of the cooling capacity of the refrigeration system, E-35. Usually, this pump will be used at lower pressures until the system is near operating temperature, when V-589 is closed and V-587 and V-549 are opened to bring the solvent circulation pump, P-35, on-line. Since the refrigeration system can handle a cooling load of 4 GPM, the use of the start-up pump, P-35, in place of the solvent circulation pump (with its capacity of 2 GPM) will reduce cool down time.

A 250-gallon polyethylene tank is used as a solvent storage or start-up tank, T-36. The complete solvent inventory can be pumped to the tank after a run to facilitate sampling, changing of the solvent system, or providing any system makeup that is required. The condition of the solvent can also be inspected visually.

Gas feed streams to the Acid Gas Removal System can be initiated from one of three sources; the gasifier, the SYNGAS system, or from a process nitrogen supply usually used in start-up situations. Feed gas from the gasifier is sent through AOV-305 and controlled at 100 psig by venting excess gas through PCV-310 to the flare. The same pressure control loop is used with a SYNGAS feed by opening V-369 and closing V-546, isolating the gasifier from the AGRS. When integrating the gasifier and AGRS, a process N_2 stream is used to pressurize the AGRS by regulating N_2 flow through PCV-308 at 100 psig. In this case, AOV-305 would be closed and V-240 open until the AGRS is ready to accept the gasifier feed. At this point AOV-305 is opened and V-240 is then closed. It should be pointed out that enough gas must be produced in the gasifier so that a portion of it can be vented for adequate pressure control at PCV-310.

The gas feed is then sent to one of a pair of dehydrating towers, D-31, Figure 8, where any residual moisture is removed by molecular sieves. A timed, mechanically-driven valve system diverts the flow to one tower while the molecular sieves in the other tower are being regenerated by heating in the presence of a metered N_2 flow, FCV-242. The pressure drop across the dehydrators is measured by DPT-315 to alert operating personnel of pluggage problems through the alarm system. The dehydrated gas is then passed through a filter, F-33, before being sent to the compressor, C-31.

The gas enters the compressor, C-31, at 100 psig, from the low pressure surge tank, T-31A. It is compressed adiabatically passing through a high pressure surge tank, T-31B, and cooled in the aftercooler, E-31, a double pipe heat exchanger with cooling water as the service fluid. Next, a knockout drum precedes the gas chiller, E-32, where solvent from circulation pump, P-35, can be used to cool the gas further. In the methanol sys-

tem, some solvent can be injected into the gas stream prior to the gas chiller, through V-1004, to prevent freezing of any residual moisture. The gas exits the gas chiller, E-32, into a surge bottle, T-33, needed to damp the pulsations of the compressor, C-31.

Compressor operation is regulated by field controllers PIC-321A and PIC-321B. Compressor suction and discharge pressures are maintained through PCV-321. In normal operation, V-1014 and V-1006 are kept closed while V-1023 is open to permit controller action. During start-up of the compressor, V-1006, the manual spillback valve, is kept open until proper compressor operation is noted.

Gas chiller operation is controlled through the operation of TCV-315. Solvent flow from the circulation pump, P-35, can be diverted to the gas chiller where TCV-315 bypasses some of the solvent flow to control gas temperature. The solvent then passes to the refrigerator, the MEA cooler or the trim heater before beginning another cycle.

The flow of compressed sour gas to the absorber, C-34, is controlled by FCV-315. Before entering the absorber, the gas can be manually sampled or monitored for oxygen content, SX-2, and composition, SX-3, with the on-line sampling equipment. The sweet gas exiting the absorber is released through PCV-345 and the flow rate is measured by FT-345. Here, also, the gas can be sampled manually or monitored by the on-line equipment, SX-4. The sweet gas is then vented to the flare for disposal.

Some of the acid gas and product gas removed in the absorber is released in the flash tank, T-35. The flash tank is first pressurized with process nitrogen through PCV-352 to approximately 140 psig. As the solvent feed to the flash tank begins releasing gas, the process nitrogen is turned off. The

objective of the flashing process is to remove any co-absorbed product gas before regenerating the solvent stream in the stripper. The flash tank gas can be sampled manually or monitored by the on-line equipment at SX-6. Flow rate is measured by FT-358 where a laminar flow element, LFE-358, is situated. The gas is then vented to the flare.

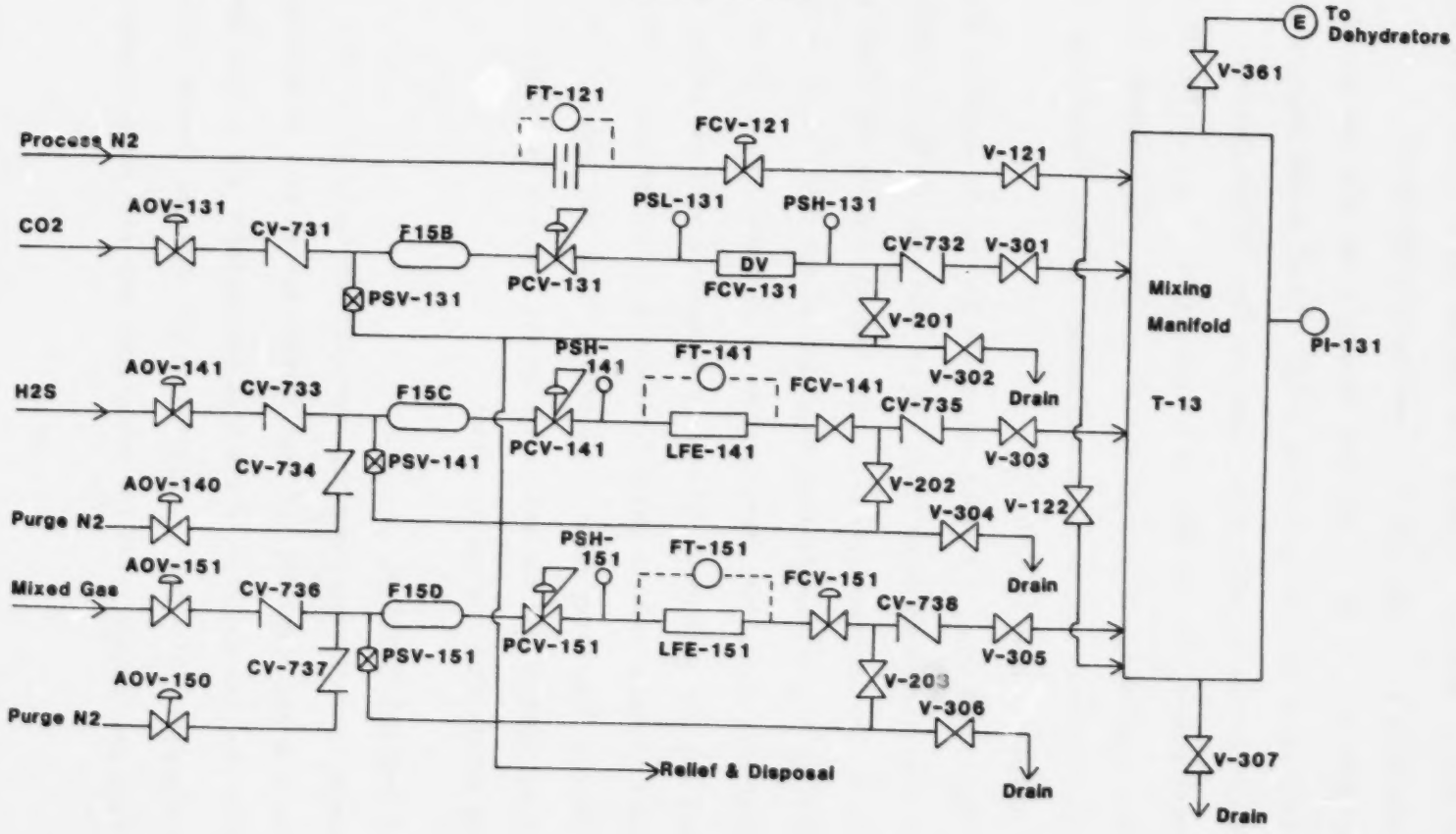
The solvent containing the acid gas is regenerated in the stripping column, C-39, where it is contacted with a stripping nitrogen stream metered in through a digital valve, FCV-393. The acid gas-N_2 mixture exits the stripper through PCV-370, before which it can be sampled manually or monitored by the on-line equipment at SX-5. The flow rate is measured by LFE-371-FT 371, and the gas is then vented to the flare.

In order to evaluate the effectiveness of each solvent cleaning system on specific gas mixtures, a synthetic gas mixing system (SYNGAS), shown in Figure 14, is used. The system is capable of blending mixtures of N_2, CO_2, H_2S, and one other gas or gas mixture before metering the feed stream to the AGRS through the dehydrators. The process nitrogen stream is metered in through FCV-121 and measured by FT-121. This stream can be diverted into both the bottom and top of the manifold, T-13, for better mixing. The CO_2 stream is supplied from a manifold of gas cylinders and directed into the mixing manifold through a digital valve, FCV-131, that can be set at the control panel. Because of the innocuous nature of these two gases, no purge system is used.

The H_2S stream is also metered into the mixing manifold from a gas cylinder through PCV-141 which is upstream of a laminar flow element, LFE-141, and PT-141. Purge N_2 is available through AOV-140 in the event of any problem. A mixed gas stream originates from a gas cylinder and its flow is measured by FT-151 in conjunction with a laminar flow element, LFE-151. Purge N_2 is

Figure 14

SYNGAS SYSTEM



available through AOV-150. Some of the planned gas mixtures to be used in this system include CO, H_2, COS, CS_2, and certain mercaptans.

The SYNGAS mix is fed to the AGRS through V-369 with the gas stream pressure controlled at PCV-310 by closing V-546. Final SYNGAS mixture composition is monitored on-line at SX-3 in the feed gas line to the absorber.

Process control for the AGRS is by ten control loops on the TDC-2000, two field controllers, and several digital valves. The solvent circulation rate is controlled by the positive displacement solvent circulation pump. The control scheme is shown in Table II.

On the liquid side, a flow of solvent is set by the positive displacement solvent circulation pump which fixes the solvent flow to the absorber. The liquid level in the absorber bottom and the liquid level in the flash tank are controlled, by regulating the flow from each vessel. As is common in level control situations, good level control causes a variable liquid flow to both the flash tank and the stripper. This in turn causes a variable amount of gas to be released in these vessels which interacts with the pressure controllers and causes the gas flow rates from the absorber, flash tank and stripper to vary. Good process control for the entire system was achieved by compromise and by accepting fairly slow controller action.

AGRS Start-up and Operation

Because current experience has been with refrigerated methanol as the solvent, start-up and operation for that solvent system will be described.

Initially, the absorber, flash tank and stripper are pressurized using process nitrogen. Nitrogen is supplied to the absorber through FCV-121 for SYNGAS runs and through V-240 for integrated runs with both streams regulated

Table II. Process Control for Gasifier-PCS System.

Control Variable	Sensor Transmitter	Control Element	Controller
Feed Gas Supply Pressure	PT-310	PCV-310	TDC-2000
Compressor Suction Pressure	PIC-321A	PCV-321	Foxboro Pneumatic Field Controllers
Compressor Discharge Pressure	PIC-321B	PCV-321	Foxboro Pneumatic Field Controllers
Sour Gas Flow Rate	FT-315	FCV-315	TDC-2000
Absorber Pressure	PT-345	PCV-345	TDC-2000
Flash Tank Pressure	PT-353	PCV-353	TDC-2000
Stripper Pressure	PT-370	PCV-370	TDC-2000
Stripper Steam Flow	TT-391	FCV-392	TDC-2000
Sour Gas Temp to Absorber	TT-315A	TCV-315	TDC-2000
MEA Temperature to Absorber	TT-349	TCV-349	TDC-2000
Absorber Liquid Level	LT-341	LCV-341	TDC-2000
Flash Tank Liquid Level	LT-351	LCV-351A LCV-351B LCV-351C	TDC-2000

by FCV-315. The pressure is initially set at 100 psig. The flash tank is pressurized through V-248 to approximately 40 psig. The stripper is set at 10 psig using FCV-393. When all vessels are at their initial set points, solvent is then circulated by P-35 or P-36 with level controllers LIC-341 and LIC-351 maintaining proper inventory in the absorber and flash tank and consequently in the stripper. The refrigeration system is then turned on with the feed temperature to the absorber set at -35°F.

After some cooling is noted, the sour gas compressor, C-31, is activated and the pressure in the absorber is brought to the desired pressure, typically 400 to 500 psig. The flash tank pressure is then reset at 100 to 200 psig. The system is allowed to cool down and the nitrogen supply to the flash tank at V-248 is closed.

When the system is operating properly and sufficient cooling is noticed, either a synthetic gas mixture or product gas from the gasifier is fed to the system, slowly replacing the initial inert gas feed. Onset of either an absorption exotherm or a desorption endotherm signals proper operation. The system is then allowed to approach steady state as marked by constant temperature, pressure, and flow rates in the system. Time to attain steady state is dependent on the solvent recirculation rate.

Experience so far has been that the system operates extremely well in both SYNGAS and integrated modes. Once the system is started up, little attention is required. As previously mentioned, there was some difficulty in coordinating control loop tuning and satisfactory mass balance closure. This was the result of very quick pressure and level control loop response to small upsets. Retuning the control loops (flash tank and stripper pressure, absorber, and flash tank level) remedied this problem allowing for adequate control while

still insuring the availability of representative gas samples. Both the start up and the steady state operation of one run, AMI-13, is presented later in this report.

Complete start up and operating procedures, in outline form, are given in Appendix I. A complete list of manual valve settings is given in Appendix II.

Alarm and Safety System

The pilot plant is equipped with an extensive alarm and automatic safety system in order to minimize the potential hazards of the operation of the facility. Two independent systems, one for the gasifier-PCS system and one for the AGRS system monitor signals from plant instrumentation for deviation from preset alarm conditions. Once an alarm condition has been detected the system initiates a switch selectable warning and/or shutdown procedure. In the case of a few critical alarm situations the entire affected system undergoes an immediate vent and purge. A list of the alarms and their available responses is shown in Appendix III.

Alarms associated with the gasifier-PCS system may initiate one of six different responses. The majority of these conditions need only activate an audible alarm (Alarms Only Mode) to warn the operator of an alarm condition. The location of the alarm is marked on the control panel by a red light. If an alarm condition is considered a dangerous one, the operator may preselect one of two other system responses. The A response calls for an immediate audible alarm followed by a two-minute time delay. If the alarm condition is not rectified within this period, all supply streams to the Gasifier-System are shut off as well as power to the utility heaters, H-13 and H-14. The B response initiates an audible alarm and an immediate shut down of supply streams and heaters. Certain selected alarms considered to be intrinsically

hazardous are hardwired to initiate a C response. This causes the gasifier and PCS system to vent and purge immediately. Loss of feed or high element temperature in either H-13 and H-14 causes an immediate shut down of electrical power to the affected heater.

The AGRS system has four alarm responses similar to the Alarm only, A, B, and C responses of the gasifier-PCS system. Most of the alarms may initiate either an audible alarm only, D, E, or F response as preselected by the operator. The E response initiates an audible alarm and an immediate shut down of the supply steams and CP-31, the sourgas compressor. The D response provides for a two-minute delay before supply and compressor shutdown and the F response immediately vents and purges the system. Several of the individual units in the AGRS system, such as the sourgas compressor and the solvent chiller, have self contained shut down systems to protect the units.

DATA ACQUISITION SYSTEM

The Data Acquisition and Laboratory computer performs two major functions. During plant operation, the system monitors plant instrumentation, providing information to the operator and creating a permanent log of the run. At times when the plant is not in operation the computer is used to maintain and reduce data from the facility and from the analytical laboratories. A block diagram of the system is shown in Figure 15.

Plant operation is monitored and regulated from a control room. Signals from 96 sensors (temperature, pressure, flow rate, etc.) are sent to a control panel, where they are processed and sent to a video display terminal and/or a Honeywell TDC 2000 process control computer and/or the data acquisition system. The TDC 2000 regulates 16 different control loops in the plant. An alarm panel superimposed on a process schematic provides visual and auditory indications of potentially hazardous conditions.

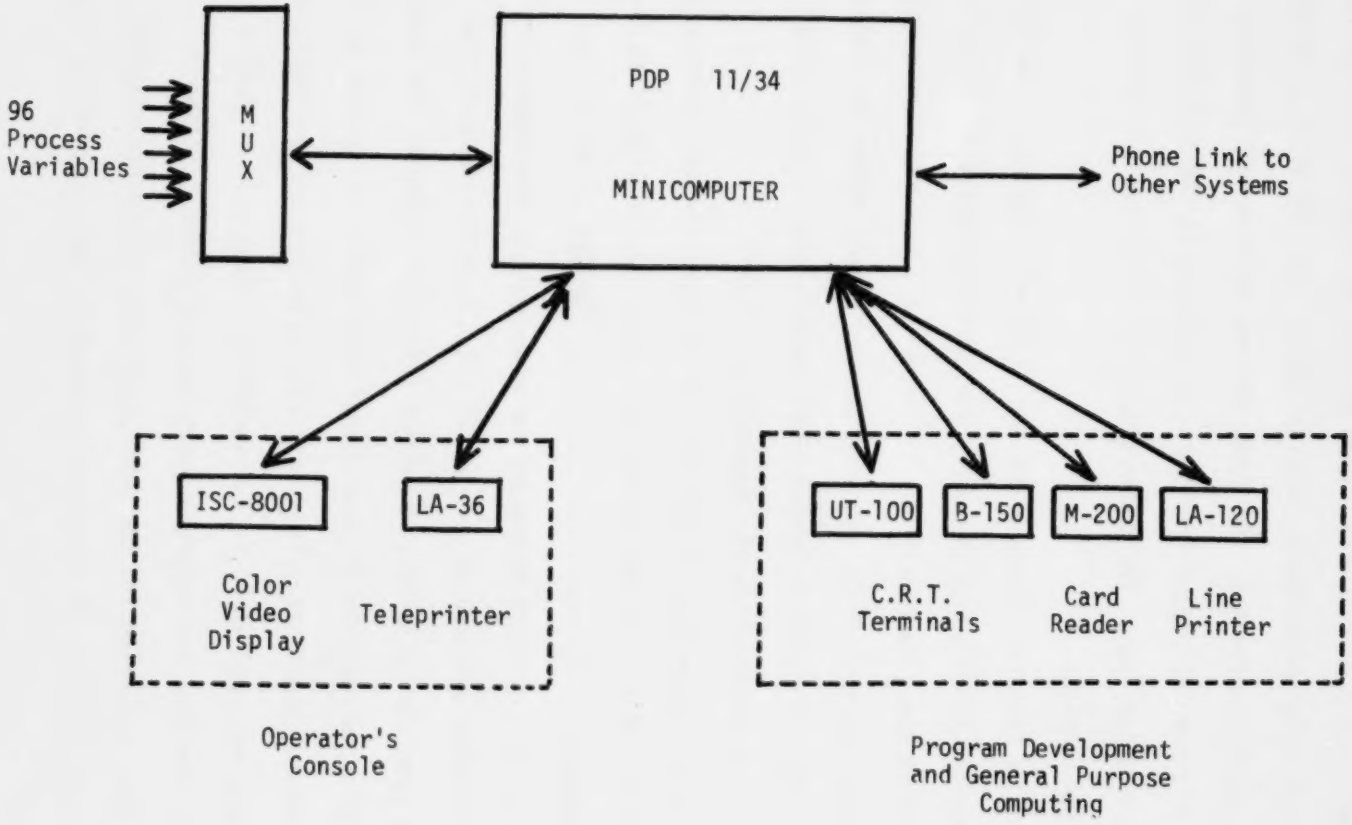


Figure 15

DATA ACQUISITION AND LABORATORY COMPUTER SYSTEM

The data acquisition system has two main objectives: to provide rapid, easily read information to the operator during plant operation, and to provide a permanent record of run data. Each process instrument is wired to a channel of an LFE Model 6100 Remote Terminal Unit. The LFE 6100, a 96 channel analog-to-digital converter, digitizes the one to five volt transmitter signals to 12 bit resolution and transmits the results to the computer through a serial communication line. The conversion takes place every 5 seconds upon command from the computer.

The system is centered around a DEC PDP 11/34 minicomputer. The 16-BIT processor supports 64,000 words of memory and 10.4 mega bytes of online disk storage. Up to 10 users terminals may be supported. Currently the general purpose facility consists of two video terminals, a card reader for input, and a fast teleprinter for output. In the plant control room, a color graphics terminal and a teleprinter form the operator's console.

The operating system used is DEC RSX-11M, an event-driven, multitasking operating system. This gives the system the capability of supporting several users simultaneously. The high level languages, FORTRAN IV and PLI are available along with PDP-11M MACRO ASSEMBLER for user program development.

The acquisition software is written in PLI. A primary acquisition task receives data from the LFE-6100 at 5 second intervals, converts the data to engineering units, and places it in a shared data region for access by the display and logging functions. This task also maintains an hour-long, circular file of data recorded at 15 second intervals. This file, called a trend-file, provides the data for interval averaging and online plotting.

During plant operation the operator views data on a video graphics terminal. Schematic displays of different sections of the plant may be viewed;

the displays resemble process flow sheets and include key plant variable values updated at 5 second intervals.

Logging takes place in two ways. A hard copy report of all system variables is available on the teleprinter at operator-selected intervals, providing an immediate permanent record of the run. The data are also saved at intervals on disk, and are later used as input to offline data reduction and processing programs.

To analyze process trends the operator may request a plot on the video graphics terminal of any of the process variables vs. time. Other functions, such as snapshotting the process and rapidly calculating mass balances, are currently being developed.

LABORATORY FACILITIES AND ANALYTICAL PROGRAM

Solid, liquid, and gas samples from the pilot plant are naalyzed in four laboratories. A summary of the analytical program is shown in Table 3. The following sections briefly describe the facilities that are used for the analyses, the equipment and instrumentation available, the sampling and sample preservation procedures, and the analytical methods used. Four laboratories have been equipped to meet the chemical analysis requirements of the project.

Laboratory Facilities

The main laboratory is a large, general purpose, laboratory in which ultimate and proximate analyses of coals and chars and all the wastewater analyses are reformed. Equipment available for these analyses include furnaces, ovens, combustion trains, and digestion and distillation racks. Whenever possible, ASTM (1) or APHA (2) guidelines have been used in the construction and the installation of the equipment.

The main laboratory also houses a water deionizer and still, several macro, semi-micro, and micro balances, glassware, reagents, and four instruments for the analysis of selected pollutants in the plant wastewater. These instruments are a Dionex System 10 ion chromatograph, an Orion Model 901 selective ionalyzer, a Dohrman DC-50 carbon analyzer, and a Baush & Lomb-Shimadzu Spectronic 210 UV-Visible spectrophotometer.

The Trace Analysis Laboratory is devoted to the analysis of trace elements by atomic absorption spectrophotometry. Instruments housed in this laboratory include a Perkin-Elmer Model 603 atomic absorption spectrophotometer with a deuterium arc and various types of flames, a Perkin-Elmer HGA-2200 graphite furnace, a Perkin-Elmer mercury analysis system, an LFE Model LTA-504 low temperature plasma asher, and a Barnstead water deionizer. A Varian Model 65 vapor generation device has been recently added.

The Coal Research and Analysis Laboratory is equipped for the study of coal pyrolysis and the analysis of sulfur, nitrogen, and free-swelling index in coals and chars. The instruments housed in this laboratory include a Fisher Scientific Model 470 sulfur analyzer, an Antek Model 707 nitrogen analyzer, a batch pyrolysis furnace, and a laminar flow reactor capable of operation at temperatures up to 1273 K with particle residence times as low as 50 milliseconds.

The Gas Chromatography Laboratory is equipped for the analyses of permanent, sulfur, and hydrocarbon gases produced in the gasification and gas cleaning process as well as liquid solvent samples. Instruments in this laboratory include a Tracor 550 gas chromatograph equipped with a thermal conductivity detector, a Tracor 550 gas chromatograph with a flame ionization detector, a Varian 3700 gas chromatograph equipped with both thermal conductivity and flame

photometric detectors, and a Perkin-Elmer Sigma X chromatography data station. A Metronix Dynacalibrator is also used for certain calibrations. Each Gas Chromatograph is equipped with both gas and liquid sampling ports. Also, provision is made for pressure-regulated injection of either hot or cold samples to facilitate the analysis of trace compounds present in the AGRS solvent.

Sampling and Sample Preservation

The sampling points throughout the plant are shown in the detailed schematic diagrams. The sampling train is shown in Figure 7. Feed coal samples are obtained from the storage drums before they are loaded into the gasifier feed hopper. The spent char is sampled from the char receiver. A sampling device is available so that samples at different depths in the receiver can be obtained corresponding to chars from the different stages of gasifier operation.

Char fines are obtained from the cyclone hopper. Each sample is then riffled and divided into three fractions. The first analysis fraction is used as received for sieve and as-received moisture analysis; the second analysis fraction is ground to pass a No. 60 U. S. Standard sieve; and the third fraction is ground to pass a No. 200 sieve. The ground samples are equilibrated to laboratory humidity overnight.

Gas samples are obtained from the gas sampling ports in heated, evacuated stainless steel bombs and in cold, evacuated stainless and glass bombs. Typically, samples are obtained from the exit of the cyclone, the sampling train, and the exit of the PCS tank. Samples of feed gas and sweet gas are obtained from the bottom and top of the absorber respectively. Samples are also collected of flash tank gas, and acid gas from the stripper.

Wastewater samples are obtained from the PCS tank and the sampling train. These samples are immediately preserved according to the procedures shown in Table 4, and kept refrigerated until analysis. Gas, methanol, and water samples are obtained from several absorber and stripper sampling ports.

Description of Analytical Methods and Procedures Used

After a plant run, the chemical analyses are performed according to a schedule such that analyses are done within maximum holding times. All wastewater holding times are shown in Table 4. Permanent gas samples can be stored for several days. Sulfur gas samples are analyzed immediately. Solid samples can be stored indefinitely. A brief description of the procedures used for analysis are given below.

Sieve Analysis--

U. S. standard sieves are used; 50 to 100 grams of sample are loaded on the top sieve and the entire assembly is shaken in a sieve shaker for 5 to 10 minutes.

AR Moisture Analysis--

ASTM-9-3173 Method is followed. Moisture is determined in the as-received sample by establishing the loss in weight of 1.0 g of sample heated to 104° to 110°C for 1 hour in a moisture oven with bone dry air circulation.

Volatile Matter Analysis--

ASTM-D-3175 method for coke is followed. Volatile matter is determined by establishing the loss in weight resulting from heating 1.0 g of sample for 6.0 minutes at 950 $\pm$ 20°C in a volatile matter furnace. Two furnaces, one controlled at 750 $\pm$ 20°C and the other at 950 $\pm$ 20°C, are available for the analysis of sparking coals.

Table 3

Analytical Program Summary

Type of Sample	Analyte	Method or Instrument
Solid	Sieve	
	AR Moisture	ASTM-D-3173
	Volatile Matter	ASTM-D-3175
	Ash	
	Carbon-Hydrogen	ASTM-D-3178
	Sulfur	Fisher Model 470 Sulfur Analyzer
	Nitrogen	Antek Model 707 Nitrogen Analyzer
Gas and Solvent	CO, H_2, N_2, CH_4, CO_2, H_2S	Varian 3700 Gas Chromotograph (TCD)
	H_2S, COS, CS_2, mercaptans, organic sulfides, thiophenes, etc.	Varian 3700 Gas Chromotograph (FPD)
	CO_2, N_2, N_2S, solvent	Tracor 550 Gas Chromotograph (TCD)
	C_1-C_5 hydrocarbons, BTX	Tracor 550 Gas Chromotograph (FID)
Wastewater	Ammonia	APHA SM No. 418
	Nitrogen	APHA SM No. 421
	Thiocyanate	APHA SM No. 413 K
	Phenolics	APHA SM No. 510 A-C
	Residue	APHA SM No. 208 A
	Sulfide	APHA SM No. 428 D
	Cyanate	APHA SM No. 413 J
	Cyanide	ASTM-D-2036
	COD	Hach COD Reactor
	TC, TOC, VC	Dohrman DC-50 Carbon Analyzer
	pH	Orion 901
	Cl^-, F^-, $SO_3^=$, $SO_4^=$	Dionex System 10 Ion Chromatograph
	Sulfur	Dionex System 10 Ion Chromatograph
	As, Be, Cd, Cr, Hg, Ni, Pb, Sb, V	Perkin-Elmer 603 Atomic Absorption Spectrophotometer

Table 4

Aqueous Sample Preservation

Analysis	Vol. Req. (ml)	Container	Preservative	Holding Time
Cyanide	500	P,G	NaOH to pH 12	24 hrs.
Cyanate	500	P,G	NaOH to pH 12	24 hrs.
Thiocyanate	500	P,G	NaOH to pH 12	24 hrs.
Sulfur	50	P,G	NaOh to pH 12	24 hrs.
Ammonia	400	P,G	H_2SO_4 to pH< 2	24 hrs.
Nitrogen	500	P,G	H_2SO_4 to pH< 2	24 hrs.
COD	50	P,G	H_2SO_4 to pH< 2	7 days
Phenolics	500	G only	H_2SO_4 to pH< 2	3 days
*Carbon	25	P,G	HCl to pH 2	24 hrs.
*TOC	25	P,G	HCl to pH 2	24 hrs.
*Sulfite	50	P,G	Formaldehyde (6 drops)	<1 hr.
*Chloride	50	P,G	None	7 days
*Sulfate	50	P,G	None	7 days
Sulfide	500	P,G	Zinc Acetate (3 drops/100ml) NaOH to pH<12 (See Note 1)	7 days
Trace Elements	500	P,G		
pH	25	P,G		6 hrs.
Residue	100	P,G	None	7 days

Note 1 Bring to pH 12 with NaOH. Add 2N Zinc acetate (3 drops/100 ml sample). Sufficient dose of zinc acetate is indicated by testing a drop of sample on lead acetate paper moistened with pH 4 acetate buffer (paper should turn black).

* Changes made for preservation procedure

Note 2 All samples stored at 4°C immediately after preservation

P = polyethylene

G = glass

Ash Analysis--

ASTM-D-3174 method is followed. The ash content is determined by weighing the residue remaining after burning 1.0 g of coal at 750°C (950°C for coke) in a muffle furnace.

Ultimate Analysis--

The feed coke and char samples ground to pass a U. S. Standard No. 60 sieve are used for C, H, and S analyses. These analyses consists of the determination of carbon and hydrogen in the gaseous products of complete combustion, the determination of sulfur, nitrogen, and ash in the material as a whole, and the calculation of oxygen by difference. The individual methods are described below.

Carbon and Hydrogen Analysis--ASTM-D-3178 method for total carbon and hydrogen in coal or coke is followed. The determination is made by burning 100 mg of sample in a combustion train and fixing the products of combustion in an absorption train after complete oxidation and removal of interfering substances.

This method gives the total percentages of carbon and hydrogen in the coal as analyzed, and includes the carbon in carbonates and the hydrogen in the moisture and in the water of hydration of silicates. Benzoic acid standards are analyzed to condition the combustion trains and to ascertain their accuracy and reproducibility before analysis of the coal and char samples.

Sulfur Analysis--Coal and coke samples are analyzed using a Fisher Sulfur Analyzer Model 470. The analyzer detects sulfur through an amperometric technique devised to measure SO_2 in the effluent produced during the combustion of sulfur-bearing materials. The analyzer has been shown to be extremely accurate

and precise in the analysis of sulfur in coal and coke. However, the analyzer does not detect sulfate sulfur which occurs in small quantities in coal. The analyzer is calibrated with certified coal standards and its calibration is checked periodically while the samples are run.

Nitrogen Analysis--Coal and coke samples are analyzed for nitrogen using an Antek Model 707 microprocessor-controlled chemiluminescence detector. The samples are pyrolyzed in a furnace and contacted with ozone. The flameless reaction between the pyrolyzate (nitric oxide) and ozone is monitored by a light sensitive detector. Nitrogen containing organic compounds and coal/coke samples analyzed by the Kjeldahl-Gunning method are used as standards.

Gas Analysis--

All gas analyses are done by gas chromatography except for the determination of steam. The concentration of steam in the gas leaving the gasifier is determined gravimetrically using the water drained from the sampling train. The fraction of gasifier exit gas passed through the sampling train is carefully metered for the calculation of the water concentration.

The pressurized gas in the sampling bombs is injected into the gas chromatographs through heat traced lines and gas sampling ports. The chromatographs are calibrated using Heise gages to introduce known amounts of calibration gases into the temperature-controlled sample loop. A Perkin-Elmer Sigma X data station is used to integrate, log, and operate on the signals from the chromatographs.

Permanent gases can be analyzed with either the Varian 3700 GC or the Tracor 550 GC each using a thermal conductivity detector. A 7 foot x 1/8 inch Carbosieve S column is used in both cases to separate H_2, N_2, CO, CH_4, H_2S and

CO_2. The same Tracor 550 equipped with a 10 foot x 1/8" SS Poropak QS column is used for the analysis of solvent samples.

Sulfur gases are analyzed for H_2S, COS, CS_2, mercaptans, organic sulfide, and thiophene using the Varian 3700 with flame photometric detector. A one foot x 1/8" Carbopak column is used for separation. Also, an 8 foot x 1/8" Chromosil 310 column is used for separation of large amounts (greater than 500 ppm) of H_2S and COS which are then analyzed with the Varian's thermal conductivity detector. Solvent samples can also be injected into the FPD to determine the amount of sulfur gases present.

Current efforts include the development of techniques for hydrocarbon analyses with the Tracor 550 FID, (C_1-C_5, Benzene Toluene Xylyene (BTX)).

Wastewater Analysis--

Wastewater samples from the sampling train may be analyzed almost immediately to obtain information on the production of pollutants. Samples from the PCS tank may be analyzed to obtain information on the buildup of pollutants from the beginning of the run until the time samples are obtained.

Sampling train wastewater samples are extremely concentrated in all pollutants because all the water originates from the condensation of the excess stream from the gasifier. The PCS wastewater samples are very dilute because 150 gallons of city water are recirculated to clean the gas throughout a run.

Ammonia--Ammonia nitrogen is determined by the distillation standard method (APHA) No. 418. The sample is buffered at a pH of 9.5 to inhibit the hydrolysis of organic nitrogen compounds, and distilled into a solution of boric acid where the ammonia nitrogen is determined acidimetrically with standard sulfuric acid, using methyl red as the indicator.

Nitrogen Analysis--Total Kjeldahl nitrogen is determined by standard Method No. 421. The nitrogen in 50 ml of sample is converted into ammonium salts by destructive digestion of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts are subsequently decomposed in a hot alkaline solution from which the ammonia is recovered by distillation and determined by acidimetric titration. A sucrose blank is always run concurrently with the samples. It should be noted that this method does not detect nitrogen in the form of oxide, azide, axo, hydrazone, nitrate, nitrite, nitrile, nitro, nitroso, oxime, and semicarbazone.

Cyanate Analysis--The cyanate content of the samples is found by determining their ammonia content before and after hydrolysis. Standard Method No. 413J is followed. The cyanate is hydrolized by heating at a low pH using sulfuric acid.

Cyanide Analysis--ASTM-D-2036 procedure is followed for this analysis. The procedure involves a distillation for concentrating and removing cyanides by refluxing the sample with H_2SO_4 and $MgCl_2$ reagent. The liberated HCN is collected in NaOH solution and its concentration is determined by a titrimetric procedure.

Chemical Oxygen Demand Analysis--The COD of the samples is determined using a Hach COD reactor. The test uses a semimicro sample digestion method. Two ml of wastewater are digested with sulfuric acid and potassium dichromate. The titration is performed with ferrous ammonium sulfate standard solution.

Thiocyanate--Standard Method No. 413 K is followed. The method entails the colorimetric determination of thiocyanate after filtration and color development with Fe $(NO_3)_3$.

Phenolics--Standard Method No. 510 A-C is used. The steam distillable phenols are reacted with 4-aminoantipyrene at a pH of 10.0 $\pm$ 0.2 in the presence of potassium ferricyanide. The solution absorbance is then determined photometrically using a Baush & Lomb-Shimadzu spectronic 210 UV/VIS spectrophotometer. This method is sensitive only to a relatively small number of phenolic compounds. A gas chromatograph with a flame ionization detector will be used in the future for a better and detailed analysis of phenolics and BTX.

Residue Analysis--Standard Method No. 208 A is followed. A well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103° to 105°C. The increase in weight over that of the empty dish represents the total residue, which is an arbitrary quantity defined by the procedure followed.

Sulfide Analysis--Standard Method 428 D is followed. Hydrochloric acid is added to the samples, after filtration, followed by excess iodine and starch indicator. The excess iodine is then back-titrated with sodium thiosulfate solution.

Total Carbon, Total Organic Carbon, and Volatile Carbon Analysis--A Dohrmann Model DC-50 carbon analyzer is used for these analyses. The instrument follows a temperature program to separate the different types of carbon; then it ozidizes the carbon to carbon dioxide. The carbon dioxide is reduced to methane and detected with a flame ionization detector.

pH Analysis--The pH is determined using an Orion Model 901 pH meter. The meter is calibrated using 4.0 and 7.0 pH buffer solutions.

Chloride, Sulfite, Sulfate, and Fluoride Analyses--The wastewater samples are analyzed for these ions in a Dionex System 10 ion chromatograph. The instrument separates the ions with an ion exchange resin and detects them with a conductivity cell.

Sulfur Analysis in Wastewater--All sulfur species in the wastewater samples are oxidized to $SO_4^=$ by heated digestion with a hot alkaline hydrogen peroxide solution. The sulfate ion is then analyzed in the Dionex System 10 ion chromatograph.

Trace Element Analysis--

All trace element analyses are done by atomic absorption analysis using a Perkin Elmer 603 Atomic Absorption Spectrophotometer, an HGA-2200 graphite furnace, a cold vapor mercury analysis system and a Varian Model 65 Vaporgenerator. Ramp heating is used for the drying and charring steps, and normal, temperature controlled, or time controlled heating is used during atomization depending on the volatility of the element. Deuterium background correction is used in all cases except in the analysis of mercury. Electrodeless discharge lamps are used in all cases except in the analysis of Cd, V, Be, and Bi where hollow cathode lamps are used.

The wastewater samples are digested by evaporative reflux with nitric acid for the analysis of all the elements of interest except mercury. Raw wastewater samples are analyzed directly for mercury. The solid samples, which have been ground to pass a No. 60 sieve and equilibrated to laboratory humidity, are prepared by either Parr bomb combustion or by low temperature ashing followed by acid bomb digestion. The liquor obtained from the oxygen bomb combustion is used for the analysis of Hg, Pb, As, Sb, and Cd. An

LFE-LTA-504 low temperature plasma asher is used for the oxidation of the samples intended for the analysis of Cr, V, B, Ni, and optionally Pb and Cd. The low temperature ashes are then digested in teflon-lined acid bombs. All acid liquors are then diluted to volume. Typical sample weight to solution volume ratios are 1 gram to 100 ml. Wastewater samples are typically concentrated from 150 to 50 ml.

Linear calibration curves are obtained, if possible, that cover the concentration ranges observed for the samples. If a sample falls out of the linear range, it is generally diluted. All analyzer parameters are optimized to give the best signal-to-noise ratio. Parameters which have been found to provide good results are listed in Table 5.

Direct calibration methods are used for the analysis of Hg, Ni, Sb, Be, Cr, and V. The method of standard additions is used for the analysis of Pb and As. Nickel complexation is used to allow high temperature charring during the analysis of As. The correlation coefficients are typically greater than 0.99 for all the calibration and standard addition lines.

The vapor generation device in an alternate form of analysis for Hg, As, and Pb when concentrations are very low or when sample matrix is not compatible to the graphite furnace analysis.

ADDITIONAL FACILITIES

In addition to the facilities mentioned previously, there are two other laboratories used to supplement project objectives:

Coal Research Laboratory

This laboratory contains both batch and laminar flow furnace reactors. These are used to study the evolution of volatile matter and trace elements

Table 5

Summary of Atomic Absorption Analysis Parameters

Element	Dry Temp-Time / Ramp Time °C-Sec / Sec	Char Temp - Time / Char Time °C-Sec / Sec	Atom Temp-Time °C-Sec	Wavelength nm	Slit nm
Cr	125-40 / 10	1100-34 / 10	2700-8	357.9	(4) 0.7
V	125-42 / 10	1700-32 / 10	2800-8	318.4	(3) 0.2
Be	125-42 / 10	1200-30 / 10	2800-8	234.9	0.7
Pb	125-40 / 10	600-34 / 10	2300-8	217.0	0.7
Ni	125-41 / 10	1000-30 / 10	2700-8	232.0	(3) 0.2
As	125-30 / 10	1100-30 / 10	2700-8	253.5	0.7
Cd	125-40 / 10	400-30 / 10	2100-8	228.8	(4) 0.7
Hg	COLD VAPOR ANALYSIS				

* Dor AA seems to be off by one nanometer

from pulverized coal during pyrolysis. Also, this enables the screening of various types of coal and char prior to use in the pilot plant.

Vapor-Liquid Phase Equilibrium Laboratory

This laboratory contains equipment for the determination of vapor-liquid equilibrium for the various solvent systems used in the AGRS. Up to this point, work has concentrated on the determination of VLE data for the methanol-CO_2-N_2-H_2S system and the development of a predictive capability from binary phase equilibrium data for the multicomponent system.

OPERATION AND RESULTS

In order to illustrate the operation of the pilot plant facility and the results which can be obtained from it, a run made on August 14, 1979 will be described. For this run the gasifier-PCS-AGRS was operated as an integrated system. The run was designated GO-28 for the gasifier-PCS system and AMI-13 for the AGRS. It consisted of the steam-oxygen gasification of Western Kentucky #11 coal char of 10x80 mesh size in the gasifier, and used refrigerated methyl alcohol as the solvent in the AGRS.

The run was one of a set designed to explore ranges of operating conditons using char in the gasifier and methyl alcohol in the AGRS. A further objective was to test the ability to maintain a steady state and to achieve closed mass balances over the entire plant. The operation and results will be described in two parts in the sections that follow.

GASIFIER-PCS SYSTEM

Operating conditions for the reactor were approximately 104 psig and an average fluidized bed temperature of 1830°F. The char feed rate was 38 lb/hr and the make gas flow rate at the PCS exit was 15.8 SCFM. The carbon conversion was 52.6%. The principal operating variables of the run, selected output variables, and material balances are shown in Table 6. Table 7 is a summary of the more important parameters of the run shown in schematic form.

Several selected process variables are shown plotted versus time in Figure 16. The two temperatures shown are those measured at 10 and 35 inches above the gas feed nozzle. Also shown is the reactor pressure

measured at the reactor top, PT-213, the product gas flow rate at the PCS exit, FT-293, and the differential pressure between two pressure taps in the fluidized bed, located 20 inches apart, DPT-21.

During the reactor startup period, 06:00 a.m. to about 11:00, several upsets occurred which resulted in excursions of bed differential pressure, temperatures, etc. Once a steady state was achieved, however, all process variables remained very steady and the system operated under automatic control without any problems.

Gas samples were taken at 14:30 for the gasifier-PCS system only and were taken for the entire system at 16:30 and 17:30. For the gasifier-PCS system, at each sample time, samples were taken at the following locations:

1. At the PCS exit, near SX-1, in stainless steel bombs at 100 psig.
2. After the cyclone, from the sample train, in stainless steel bombs at 100 psig.
3. After the cyclone, from the sample train, in glass bombs at 20 psig. These bombs are mainly for sulfur gas analysis.

The residence time of the reactor-cyclone, PCS tank system is such that after approximately 90 minutes, the gas composition from samples taken at the sample train and the PCS exit are essentially identical. The experimental time constant for the PCS tank at 100 SCFM is 24 minutes. From Figure 16 it can be seen that steady state conditions were achieved at about 11:00. The values of the process variables used in Tables 6 and 7 are averages over the period 12:30 to 17:30. The gas analysis shown in the tables is the average of the three samples times noted on Figure 16.

As can be seen from Table 6, the mass balance closures for total mass and the major elements is excellent and indicates that flow rate measurements

Table 6

NCSU DEPARTMENT OF CHEMICAL ENGINEERING

FLUIDIZED BED COAL GASIFICATION REACTOR

RUN GO-28 8/14/79 12:30-17:30

REACTOR SPECIFICATIONS

PRESSURE = 104.0 PSIG (818.4 KPA)
TEMPERATURE = 1830.0 DEG.F (998.9 DEG.C)
BED HEIGHT = 38.0 IN. (0.97 METERS)
BED DIAMETER= 6.0 IN. (0.152 METERS)
ESTIMATED BED VOIDAGE = 0.75
BED EXPANSION FACTOR = 1.65
ESTIMATED LEAK RATE = 0.79 SCFM

SOLID FEED PROPERTIES

WESTERN KENTUCKY #11 COAL CHAR, 10X80 MESH
PARTICLE DENSITY = 112.2 LB/FT**3 (1.797 G/CM**3)
SETTLED BED DENSITY = 46.2 LB/FT**3 (0.740 G/CM**3)
AVERAGE PARTICLE DIAMETER = 493.85 MICRONS
A-R MOISTURE CONTENT = 0.0108

PROXIMATE ANALYSIS OF COAL FEED

FIXED CARBON	= 0.839
VOLATILE MATTER	= 0.022
MOISTURE	= 0.011
ASH	= 0.127

ULTIMATE ANALYSES

	COAL FEED	SPENT CHAR	CYCLONE DUST
CARBON	0.813	0.726	0.624
HYDROGEN	0.004	0.002	0.003
OXYGEN	0.018	0.015	0.009
NITROGEN	0.010	0.001	0.010
SULFUR	0.026	0.016	0.015
ASH	0.127	0.240	0.340

FEED RATES

COAL = 38.16 LB/HR AT 200.0 DEG.F
= 17.31 KG/HR AT 93.3 DEG.C

STEAM = 59.59 LB/HR AT 365.3 DEG.F
= 27.03 KG/HR AT 185.2 DEG.C

OXYGEN = 16.26 LB/HR AT 84.4 DEG.F
= 7.37 KG/HR AT 29.1 DEG.C
= 3.04 SCFM

NITROGEN = 4.55 LB/HR AT 87.8 DEG.F
= 2.06 KG/HR AT 31.0 DEG.C
= 0.97 SCFM

PURGE N2 = 5.82 LB/HR (1.24 SCFM)
= 2.64 KG/HR

FEED RATIOS AND CONDITIONS

STEAM/COAL = 1.81 LB STEAM/LB COAL(MAF)
= 1.28 MOLES STEAM/MOLE CARBON

O2/COAL = 0.49 LB O2/LB COAL(MAF)
= 0.20 MOLES O2/MOLE CARBON

N2/O2 = 0.32 MOLES N2/MOLE O2

COMBINED GAS FEED TEMPERATURE = 990.0 DEG.F
= 532.2 DEG.C

SUPERFICIAL GAS VELOCITY IN REACTOR = 1.17 FT/S
= 0.36 M/S

V(MIN.FLUID.) = 0.211 FT/S (6.4 CM/S)
VS/V(MIN.FLUID.) = 5.5

Table 6 Continued

RUN GO-28 8/14/79 12:30-17:30

CONTROL VARIABLES

TEMPERATURE	= 1830.0 DEG.F
N2/O2 MOLAR FEED RATIO	= 0.32
STEAM PARTIAL PRESSURE	= 98.69 PSI
BED HEIGHT	= 3.17 FT
SOLID SPACE TIME	= 27.3 MIN
GAS SPACE TIME	= 2.71 S

OUTPUT VARIABLES

PRESSURE DROP OVER 20 IN.	= 9.0 IN.H2O (0.325 PSI)
	= 2.2 KPA
PCS GAS FLOW RATE	= 2.64 LB-MOLE/HR (15.79 SCFM)
	= 2.62 LB-MOLE/HR(DRY BASIS)
CYCLONE GAS FLOW RATE	= 4.92 LB-MOLE/HR (29.46 SCFM)
	= 2.62 LB-MOLE/HR(DRY BASIS)
	= 2.23 KMOL/HR
SOLID HOLDUP	= 17.3 LB (7.9 KG)

PRODUCT FUEL PROPERTIES

CO = 21.7% (MOLAR BASIS)
= 0.446 LB CO PRODUCED/LB COAL(MAF)
H2 = 36.2% (MOLAR BASIS)
= 0.054 LB H2 PRODUCED/LB COAL(MAF)
CH4 = 2.7% (MOLAR BASIS)
= 0.031 LB CH4 PRODUCED/LB COAL(MAF)

HEATING VALUE OF MAKE GAS	= 3473.3 BTU/LB
	= 225.8 BTU/SCF
	= 8072.2 KJ/KG
HEATING VALUE OF SWEET GAS	= 8714.3 BTU/LB
	= 332.4 BTU/SCF
	= 20252.7 KJ/KG

CONVERSION VARIABLES

CARBON CONVERSION	= 52.6%
STEAM CONVERSION	= 31.1%
SULFUR CONVERSION	= 59.0%

SOLID MATERIAL BALANCE

COAL FED	= 383.0 LB
SPENT CHAR COLLECTED	= 211.5 LB = 55.2% OF FEED
CYCLONE DUST COLLECTED	= 21.0 LB = 5.5% OF FEED
COAL GASIFIED	= 150.5 LB = 39.3% OF FEED
SPENT CHAR REMOVAL RATE	= 16.0 LB/HR
CHAR RATE FOR MASS BALANCE	= 19.1 LB/HR

	CYCLONE EXIT GAS ANALYSIS			PCS EXIT GAS ANALYSIS		
	%WET	%DRY	LBMOL/HR	%WET	%DRY	LBMOL/HR
CO	10.64	19.96	0.524	19.85	19.96	0.524
H2	17.77	33.33	0.875	33.16	33.33	0.875
CH4	1.30	2.44	0.064	2.43	2.44	0.064
CO2	15.37	28.83	0.757	28.67	28.83	0.757
N2	7.86	14.75	0.387	14.67	14.75	0.387
H2S	0.36	0.67	0.018	0.67	0.67	0.018
COS	0.01	0.03	0.001	0.02	0.03	0.001
H2O	46.68		2.298	0.53		0.014

H2S = 6692.0 PPM(DRY)
COS = 250.0 PPM(DRY)

Table 6 Continued

RUN GO-28 8/14/79 12:30-17:30

ELEMENTAL MATERIAL BALANCES : FLOWS IN LB/HR

	MASS	C	H	O	N	S
COAL	38.2	31.04	0.17	0.70	0.40	1.002
GASES	86.2	0.00	6.67	69.17	10.37	0.000
TOTAL INPUT	124.4	31.04	6.84	69.87	10.76	1.002
CHAR	16.0	11.63	0.03	0.25	0.01	0.256
DUST	1.6	0.99	0.00	0.01	0.02	0.023
GASES	103.7	16.16	6.69	69.37	10.84	0.584
WASTEWATER	0.0	0.00	0.00	0.00	0.00	0.000
TOTAL OUTPUT	121.3	28.78	6.72	69.63	10.87	0.863
% RECOVERY	97.5%	92.7%	98.3%	99.7%	101.0%	86.2%

FEED AND EFFLUENT TEMPERATURES

COAL IN = 200.0 DEG.F
GASES IN = 990.0 DEG.F

CHAR OUT = 1830.0 DEG.F
GASES OUT = 1716.0 DEG.F

ENERGY BALANCE

ENERGY IN = -3.02E+05 BTU/HR (-3.18E+05 KJ/HR)
ENERGY OUT = -3.10E+05 BTU/HR (-3.27E+05 KJ/HR)
HEAT LOSS = 8.50E+03 BTU/HR (8.96E+03 KJ/HR)

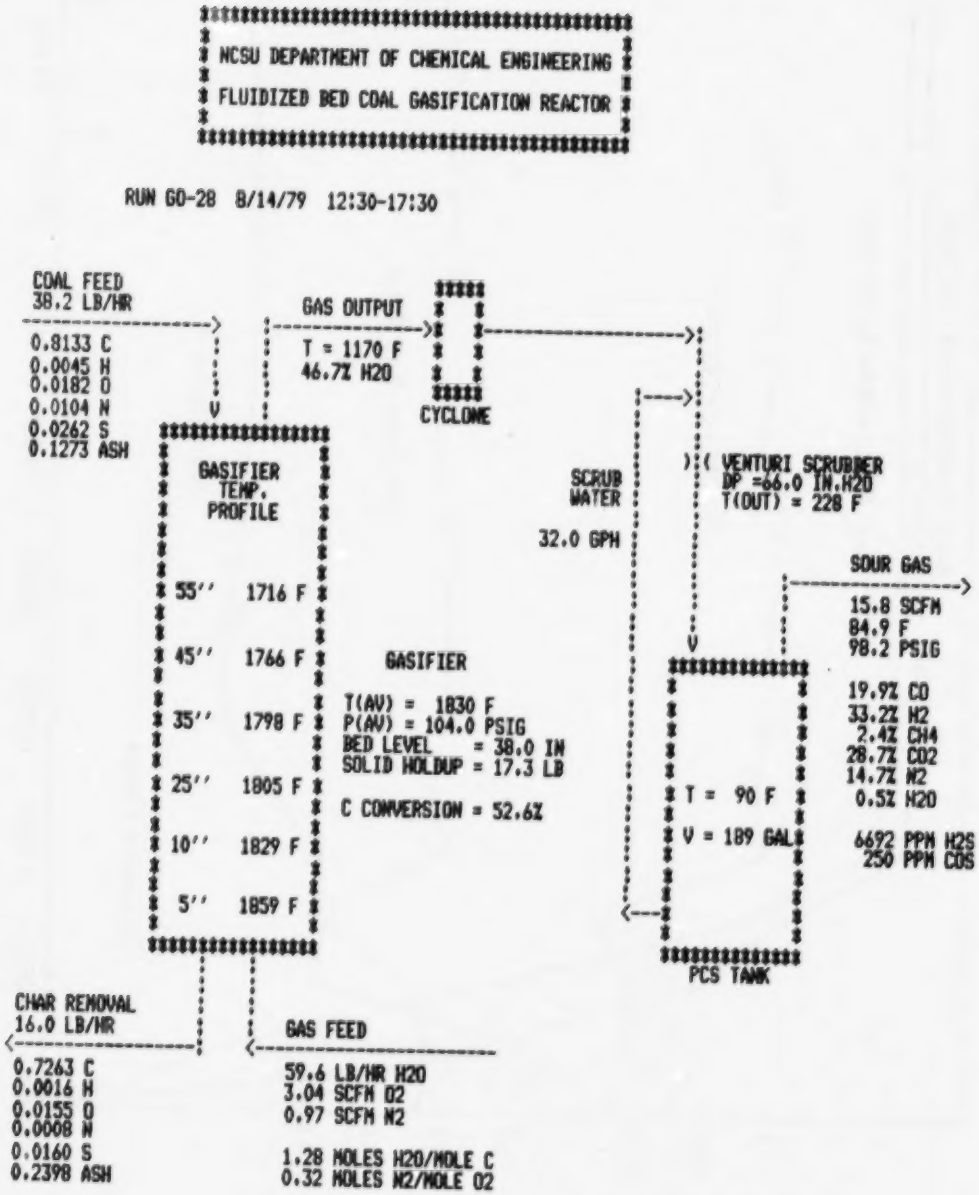
% HEAT LOSS = 2.8%

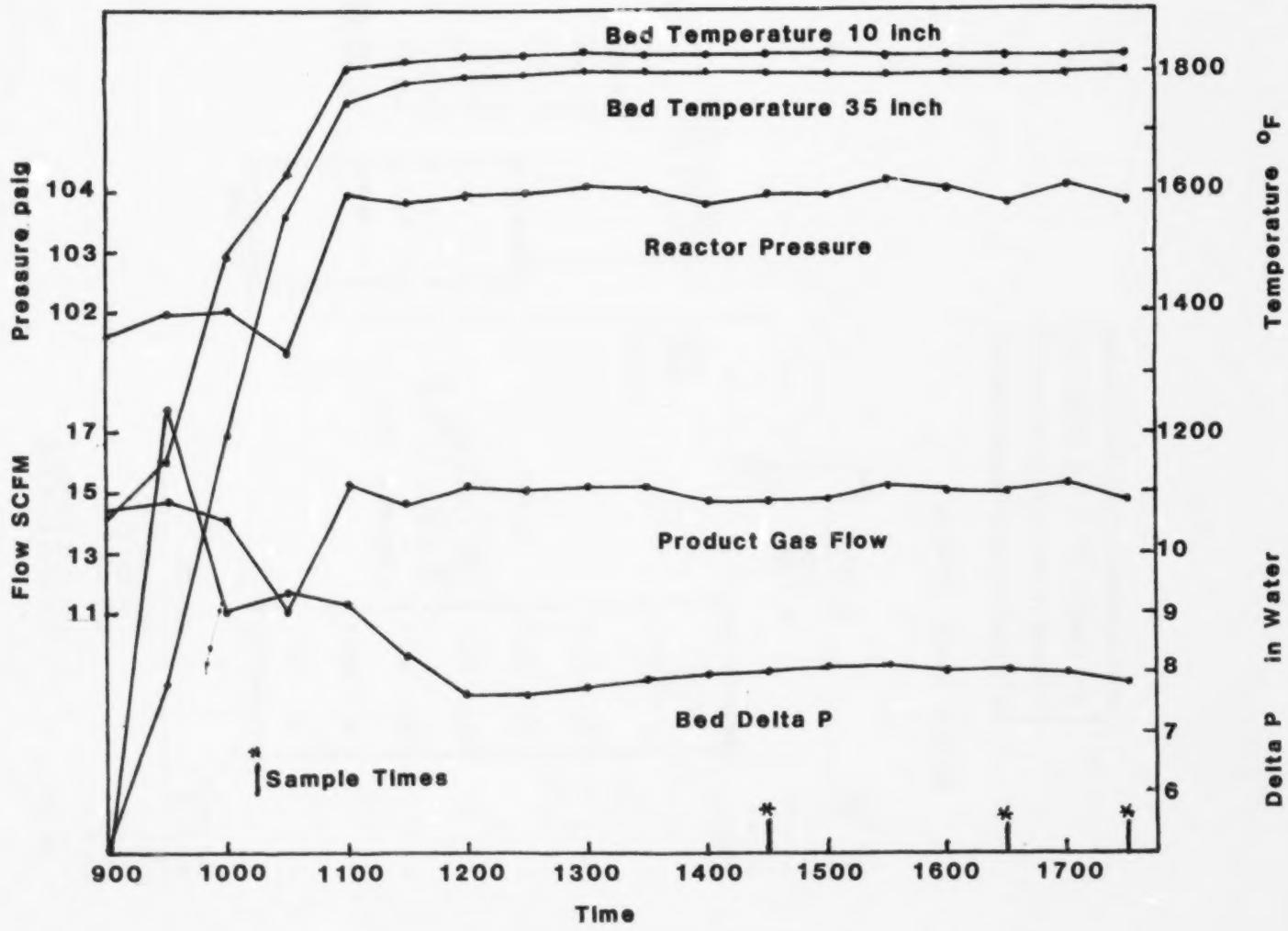
TRAP WATER ANALYSIS

PH = 6.89

SPECIES	C(MG/LITER)	PRODUCTION RATE IN GASIFIER		LB PRODUCED/LB COAL FED
		LB/HR	MG/HR	
NH3	1335.00	5.53E-02	2.51E+04	1.45E-03
CL	8.33	3.45E-04	1.56E+02	9.04E-06

Table 7


NCSU DEPARTMENT OF CHEMICAL ENGINEERING
FLUIDIZED BED COAL GASIFICATION REACTOR
RUN GO-28 8/14/79 12:30-17:30
COAL FEED
38.2 LB/HR
0.8133 C
0.0045 H
0.0182 O
0.0104 N
0.0262 S
0.1273 ASH
GAS OUTPUT
T = 1170 F
46.7% H2O
CYCLONE
GASIFIER
TEMP.
PROFILE
55'' 1716 F
45'' 1766 F
35'' 1798 F
25'' 1805 F
10'' 1829 F
5'' 1859 F
GASIFIER
T(AV) = 1830 F
P(AV) = 104.0 PSIG
BED LEVEL = 38.0 IN
SOLID HOLDUP = 17.3 LB
C CONVERSION = 52.6%
SCRUB
WATER
32.0 GPH
VENTURI SCRUBBER
DP =66.0 IN.H2O
T(OUT) = 228 F
SOUR GAS
15.8 SCFM
84.9 F
98.2 PSIG
19.9% CO
33.2% H2
2.4% CH4
28.7% CO2
14.7% N2
0.5% H2O
6692 PPM H2S
250 PPM COS
T = 90 F
V = 189 GAL
PCS TANK
CHAR REMOVAL
16.0 LB/HR
0.7263 C
0.0016 H
0.0155 O
0.0008 N
0.0160 S
0.2398 ASH
GAS FEED
59.6 LB/HR H2O
3.04 SCFM O2
0.97 SCFM N2
1.28 MOLES H2O/MOLE C
0.32 MOLES N2/MOLE O2



GASIFIER - PCS RUN GO-28

Figure 16

and gas analyses are accurate. The closure for sulfur is not as good but is expected to improve when sulfur in the waste water is properly accounted for.

A kinetic model of the gasifier, based on kinetics developed by Johnson (3), was programmed for the data acquisition system computer. The model takes as input the reactor average bed temperature and pressure, feed rates of coal, steam, oxygen, nitrogen, and purge nitrogen, solids holdup, ultimate analysis of the feed coke and spent char, the relative reactivity of the coke and the CO/CO_2 distribution coefficient.

The model output, consisting of one page, echos the reactor specifications and feed rates and shows the comparison of the model and experimental values for dry make gas flow rate, char removal rate, carbon conversion and gas composition (wet basis). Table 8 shows the results of the model predictions for GO-28. For this run, the prediction is very good.

In general, for those runs with good mass balances and good fluidized beds the model compares well with experimental results.

A full report on the results of the first set of planned gasifier runs is to be issued soon.

ACID GAS REMOVAL SYSTEM

As was indicated, the gas produced in the gasifier during GO-28 was fed to the AGRS where it was contacted with refrigerated methanol to remove CO_2 and sulfur gases. The operating conditions for this run are represented schematically in Figure 17. It should be mentioned that in both the stripping and absorption columns 21.3 feet of 1/4-inch ceramic Intalox saddles were used as the packing material.

Table 8

WELL-MIXED CHAR GASIFICATION
MODEL RESULTS

GO-28 8-14-79 12:30-17:30

REACTOR SPECIFICATIONS

BED PRESSURE(ATM)	104.00
BED TEMPERATURE(F)	1830.00
SOLIDS HOLDUP(LB)	17.30
BED HEIGHT(IN)	38.00
BED DIAMETER(IN)	6.00

FEEDRATES(LB/HR)

INLET CHAR	38.16
STEAM	59.59
OXYGEN	16.26
NITROGEN	4.55
HYDROGEN	0.00
PURGE N2	5.82

MODEL PARAMETERS

PRETREAT TEMP(F)	2000.00
CHAR REACTIVITY	0.4030
COMBUSTION EXTENT	0.8030

FEED CHAR ANALYSIS(WT PERCENT)

CARBON	81.30
HYDROGEN	0.40
OXYGEN	1.80
NITROGEN	1.00
SULFUR	2.60
ASH	12.70

RESULTS

	MODEL	EXPERIMENTAL
DRY GAS FLOW RATE (SCFM)	16.24	15.79
STEAM CONVERSION	0.335	0.311
CARBON CONVERSION		
COMBUSTION	0.245	
GASIFICATION	0.333	
TOTAL	0.578	0.526
ASH CONTENT OF CHAR	25.63	24.00
CHAR REMOVAL RATE (LB/HR)	18.04	16.00

GAS COMPOSITION (MOLE PERCENT)

	MODEL	EXPERIMENTAL
CO	11.39	10.64
H2	17.00	17.77
CH4	2.94	1.30
CO2	16.08	15.37
N2	7.62	7.86
H2S	0.21	0.36
COS	0.00	0.01
H2O	44.76	46.68

RUN NUMBER A-M-13
INTEGRATED RUN
DATE 8/14/1979

COLUMN TEMPERATURE PROFILES & MASS BALANCES

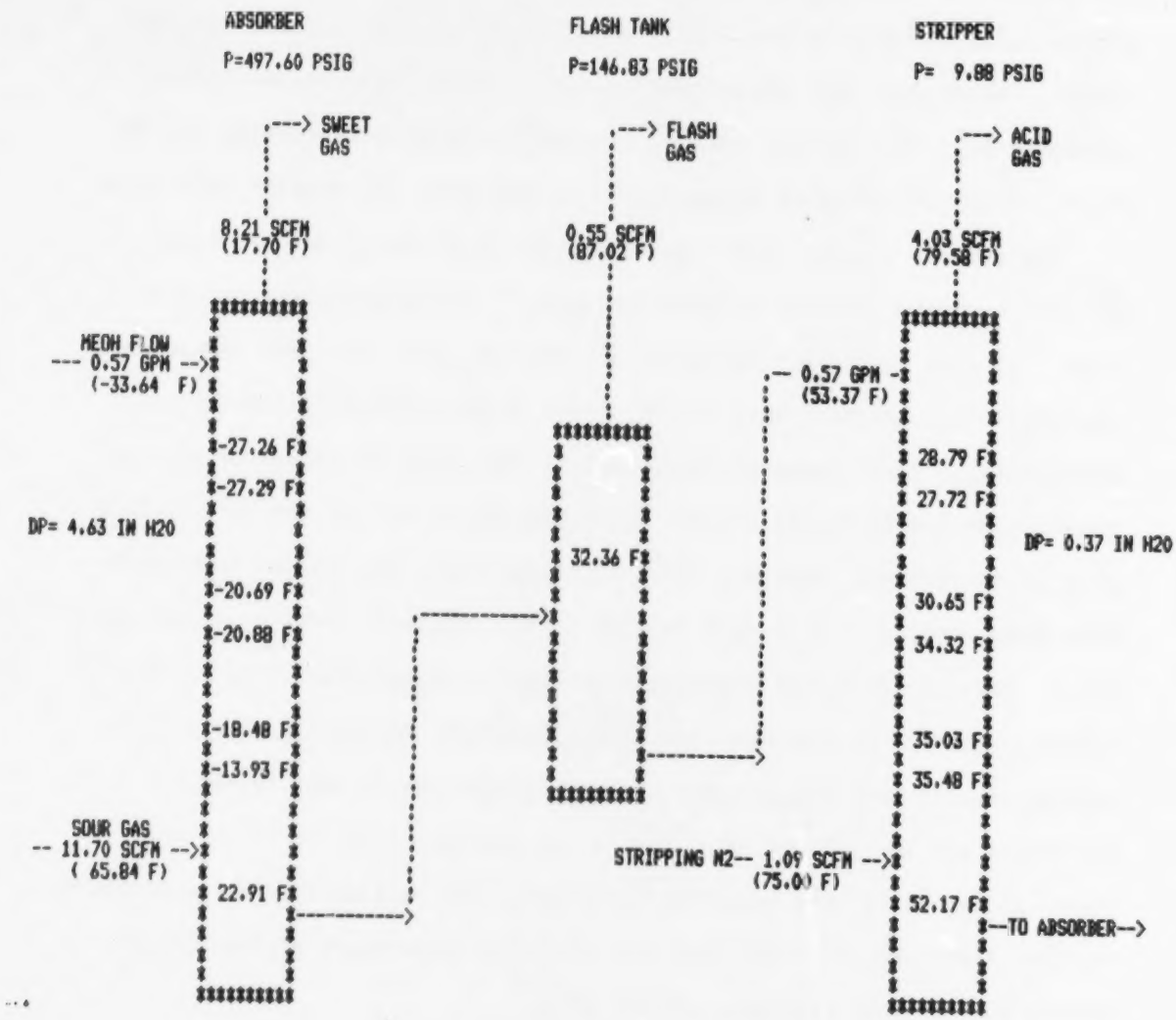


Figure 17

Several process temperatures are depicted in Figure 18. Normal operating procedure calls for the system to be cooled down using refrigerated methanol with only N_2 as the feed gas to the absorber. When gasifier operation has stabilized, the product gas is then fed to the absorber. During AMI-13, system integration took place at 11:30 and can be seen in the temperature rise of the solvent in the absorber bottoms reservoir. This, of course, is attributed to the heat of solution of the acid gases in the methanol. After some lag, the desorption endotherm in the stripper is also evident. At 17:30, changes seen in the column temperature profiles are the result of the beginning of system shut down and after all samples were taken.

The gas and liquid samples were taken at 16:30 and 17:30. Averages of all process parameters and analyses are used in the mass balance calculations. It should be noted that while the absorber and flash tank temperature are fairly constant over this period, there is some change in the stripper temperatures. These temperature variations stem from the amount of solvent inventory in the system at a given time, but, since most of the solvent hold up is in the bottoms reservoir of the stripper where the solvent is essentially clean methanol, it is felt that this has a minimal effect on system operation. This, in fact, has been substantiated by several runs using a CO_2-N_2-methanol system. Furthermore, the slow temperature decline in the stripper packing results from the exchange of heat between the solvent having the stripper bottom and the solvent being fed to the stripper from the flash tank. (Refer to Figure 10, the Lean/Rich Exchange). The packing section temperature profile, therefore, will not line out until the temperature of the solvent leaving the stripper reaches a steady state.

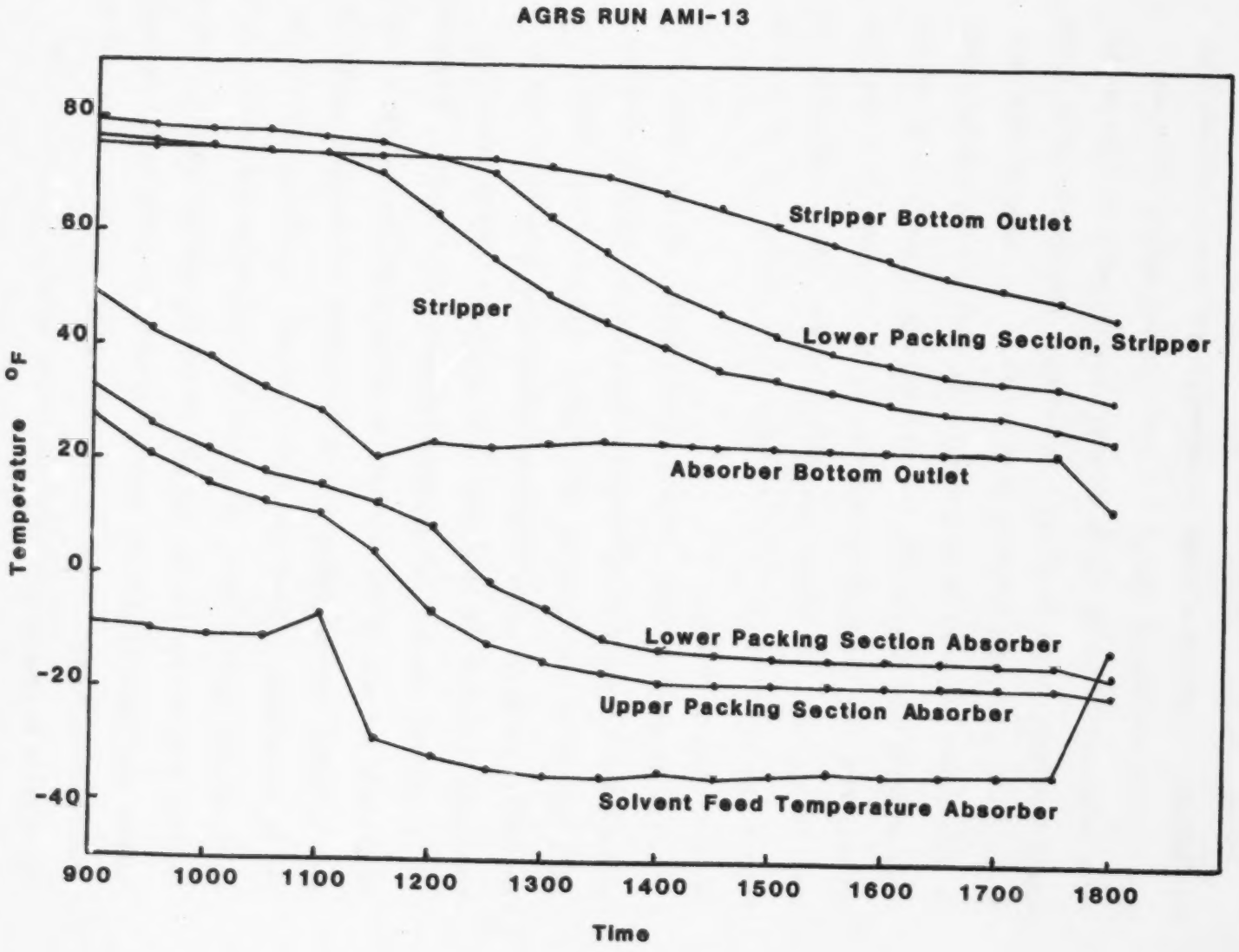

AGRS RUN AMI-13
80
60
40
20
0
-20
-40
Temperature °F
900
1000
1100
1200
1300
1400
1500
1600
1700
1800
Time
Stripper Bottom Outlet
Stripper
Lower Packing Section, Stripper
Absorber Bottom Outlet
Lower Packing Section Absorber
Upper Packing Section Absorber
Solvent Feed Temperature Absorber

Figure 18

The mass balance calculations for AMI-13 are presented in Table 9. Mass balance closure was adjusted to compensate for several problems that were found at the time of the run. A leak of approximately 1 SCFM was found in the relief system in the absorber, and the sweet gas flow was adjusted to compensate for this loss. In addition, process controller tuning caused fairly wide oscillations in acid gas flow. The methanol flow rate to the stripper was found to be the reason for this problem. Because this steam contains H_2S, CO_2, and COS, the concentration of the gases in the acid gas will vary with the inlet solvent flow to the stripper. To account for this, the CO_2, H_2S, and COS balances were adjusted. The problems with COS balance closure can be attributed to analytical inaccuracies. The problem has since been corrected.

Also presented in Table 9 are calculated solvent stream compositions. Although the streams were sampled and analyzed, there are still problems in these areas which are now being investigated. The calculated compositions are checked using the vapor-liquid equilibrium data generated in the VLE laboratory, assuming the flash tank is an adiabatid flashing operation.

In general, the AGRS has operated extremely well. Usually, before any adjustments are made in the mass balance calculations, better than 92% closure is found. Current emphasis is on developing a mathematical model for the CO_2-N_2-methanol systems with plans to extend this calculation for the multicomponent system. Also, since less than a tenth of the available mass transfer area is required for the bulk of the CO_2 and H_2S removal in the absorber, additional temperature sensors are being installed to follow mass transfer rates by monitoring the absorption exotherm. It is hoped that this, in addition to samples taken from the column packing, will provide the

Table 9

NCSU DEPARTMENT OF CHEMICAL ENGINEERING

ACID GAS REMOVAL SYSTEM

RUN NUMBER A-H-13
INTEGRATED RUN
DATE 8/14/1979

STREAM COMPOSITION (VOL %)

	SOUR GAS	SWEETGAS	FLASHGAS	STRIPN2	ACID GAS	ABSORBOT*	FLASHBOT*	STRIPBOT*
CO2	29.100	0.000	48.070	0.000	77.890	7.374	6.847	0.004
H2S	0.658	0.000	0.290	0.000	1.870	0.167	0.164	0.000
COS	0.044	0.000	0.077	0.000	0.147	0.011	0.010	0.000
MEOH	0.000	0.000	0.000	0.000	0.000	92.290	92.903	99.442
H2	33.870	48.990	23.590	0.000	0.000	0.000	0.000	0.000
CO	20.030	28.300	15.900	0.000	0.000	0.045	0.000	0.000
N2	13.730	19.930	8.720	100.000	19.590	0.000	0.000	0.521
CH4	2.400	2.780	3.350	0.000	0.490	0.114	0.075	0.034

* CALCULATED

MASS BALANCE (LB-MOLES/HR)

	IN		OUT					
	SOUR GAS	STRIP N2	SWEETGAS	FLASHGAS	ACID GAS	TOTAL IN*	TOTAL OUT*	% RECOVERY
CO2	0.569	0.000	0.000	0.044	0.525	0.569	0.569	99.953
H2S	0.013	0.000	0.000	0.000	0.013	0.013	0.013	99.961
COS	0.001	0.000	0.000	0.000	0.001	0.001	0.001	123.295
MEOH	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
H2	0.662	0.000	0.672	0.022	0.000	0.662	0.694	104.742
CO	0.392	0.000	0.388	0.015	0.000	0.392	0.403	102.846
N2	0.268	0.182	0.273	0.008	0.132	0.451	0.413	91.723
CH4	0.047	0.000	0.038	0.003	0.003	0.047	0.045	94.847
TOTAL (LB-MOLES/HR)	1.955	0.182	1.372	0.092	0.674	2.134	2.137	100.129

* METHANOL-FREE BASIS

TOTAL METHANOL LOSS= 0.000 LB-MOLES/HR = 0.000 GALLONS/HR

necessary information to characterize the solvent system used.

REFERENCES

1. American Society for Testing and Materials, 1976 Annual Book of ASTM Standards

2. American Public Health Association, American Water Association, and Water Pollution Control Federation, Standard Methods for the Examination of Water and Wastewater, 14th ed., Washington, D. C., American Public Health Assoc., 1976.

3. Johnson, J. L., "Kinetics of Bituminous Coal Char Gasification, with Gases Containing Steam and Hydrogen," Adv. Chem. Ser., No. 131, 1974.

4. Ferrell, J. K., R. W. Rousseau, and D. G. Bass, "The Solubility of Acid Gases in Methanol," EPA-600/7-79-097, April, 1979.

5. Agreda, V. H., R. M. Felder, and J. K. Ferrell, "Devolatilization Kinetics and Elemental Release in the Pyrolysis of Pulverized Coal," EPA-600/7-79-241, November, 1979.

APPENDICES

Appendix I

GASIFIER - PCS PRESSURE TEST

The pressure test should be made the day before each gasifier run to determine the leak rate for use in the material balance calculations and to assure that the system has no major gas leaks.

A leak rate of less than 0.80 SCFM has proven to be satisfactory.

1. After the vessels are sealed set PIC-310 in auto at 100.0 psig
2. Open AOV-116 and set FIC-116 (N_2) at 12.0 SCFM
3. Open AOV-101 and set all purges at a low value (~10)
4. Check that the valve to the on-line sample port SX-1 is closed
5. When gasifier reaches about 75 psig manually check all fittings (with snoop) that have been opened since the last run
6. Open the valve to the cyclone sample train and set a flow of about 4 at the rotameter. Check all fittings (especially the seal around the filter housing) for leaks. Close inlet valve to close off cyclone sample train.
7. Continue pressurization until reactor is at 100 psig
8. Close all purge N_2 valves and close AOV-101
9. Start Data Acquisition System. Run (300,2) PTEST
10. Put FIC-116 in loop manual and close the control valve; close AOV-116
11. Put PIC-310 in loop manual and close the control valve
12. Let the pressure decay for at least 30 min.
13. Turn off data acquisition system; Run Stop .

14. Calculate the pressure decay

    A. Choose two times about 10 min. apart (start about 5 min. after start of pressure decay)

    B. Substract pressure readings (channel 25) and divide by time span

    C. This gives psi pressure drop/min (ΔP)

15. Calculate the system volume from following equation

$$V\ (ft^3) = 120.9 - \frac{\#\ \text{coke in hopper}}{46.2} - \frac{\text{PCS tank level} + 16}{12}\ 7.07$$

16. Calculate the leak rate from the following equation:

$$\text{Leak rate (SCFM)} = \frac{\Delta P \times V}{10.73 \times 535} \times 359$$

NUCLEAR LEVEL GAUGE CALIBRATION

LT-101, REACTOR BED LEVEL INDICATOR

The Texas Nuclear density gauge is to be calibrated so that the recorder indicates 10% when the reactor is empty, and 90% wher a 1/2-inch steel plate is inserted in front of the source. The radiation beam is about two inches in diameter, and the recorder goes from 10% to 90% as the top of the coal bed passes the beam.

Calibrate the level gauge while the reactor is empty. Open the shutter.

1. Turn on the control panel switch and allow the detector to warm up for 24 hours, if not already on.
2. Connect a chart recorder to the gauge output in order to determine that an indication has stabilized.
3. Set the meter and recorder at 10% with the "baseline position" control.
4. Hang the 1/2-inch steel plate between the source and the reactor vessel. Wait about 5 minutes between steps.
5. Adjust the "density calibration" control to make the output read 90%.
6. Remove the steel plate. If the recorder does not indicate 10%, repeat steps 3 through 6.
7. Close the radiation source shutter. The recorder will go off up-scale.

ON-LINE EQUIPMENT SET-UP

O_2 Monitor

1. Remove cell holder from mount
2. Check that range is on high
3. Adjust to "cal" point with span adjustment
4. On lower right hand side of SX-1 cabinet hook-up vent line
5. Open V-611 on panel
6. Open "sample" valve on SX-1 O_2 meter
7. Adjust flow to approx. 1 SCFH
8. When reading is less than 5% on 0-25% scale put range on MED

CO & CO_2 Analyzers

1. Open main valve on CO, CO_2 calibration tank
2. Set V-651 on N_2
3. Adjust "zero" on both meters
4. Set V-651 on CO/CO_2
5. Adjust gain on both meters to calibration gas concentration
6. Repeat 2 to 5 until zero and range both correct
7. Set V-651 on N_2; close main valve on gas cylinder
8. Open SX-1 (at control panel)
9. Close V-617
10. Should observe approximately 26-28 psi pressure
11. If not open valve on SX-1 panel at filter outlet
12. Set all cabinet purges to 10 SCFH
13. Turn all dryers on
14. Set-up recorders in control room
15. If you wish to look at port other than SX-1
    A. Close V-611 (O_2 monitor stays at SX-1)

B. Turn on SX- port that is desired at control panel

C. Open appropriate valve on panel (in plant)

D. Change as desired

E. If stripper exit is desired turn pump on first

Cyclone Sample Train

1. Turn on heat tracing of sample train line
2. Turn on cooling water to sample train condenser
3. After startup is nearly complete set sample train floor at a rotameter reading of 8.

SHUT-DOWN FOR ON-LINE EQUIPMENT

CO & CO_2 analyzers

None required

O_2 monitor

1. Wait until all AOV's on gasifier are in manual
2. Turn off SX-1 at control panel
3. Open V-617; set range on high
4. Open V-611 (may already be open)
5. Set V-651 on N_2 (may already be there)
6. Turn cabinet purges off
7. Turn dryers off
8. Close sample valve on SX-1 O_2 monitor
9. Hook-up CO_2 cylinder to "span" fitting
10. Open "span" valve
11. Set CO_2 pressure at approximately 10 psig
12. Let flow for approximately 10 minutes at 2 SCFH
13. Remove vent line
14. Install plugs and turn "span" valve off at the same time
15. Remove CO_2 cylinder

Cyclone Sample Train

Allow sample train to run during shut down for a sufficient time to purge test meter with nitrogen.

1. Turn off sample train flow
2. Turn off cooling water
3. Turn off heat tracing
4. Drain remaining water from condensate tank

GASIFIER OPERATION PROCEDURE

Day before Run

1. Complete pre start-up checklist (See appendix)
2. Turn on nuclear level gauge.
3. Run pressure test

Day of Run

1. Turn exhaust fans on
2. Light flare
3. Check status of on-line equipment
4. Reset alarm system at alarm panel
5. Set AOV's according to following table

AOV	Setting	AOV	Setting
110	Auto	101	Auto
111	Closed	213	Auto
115	Auto	215	Auto
116	Auto	241	Closed
117	Auto	254	Auto
		270	Auto
		294	Auto
		296	Auto
		297	Auto

6. Check that PIC-310 is in auto at 99.5 psig
7. Set purges at a low value (approximately 10 SCFH)
8. Set FIC-116 at 12 SCFM; turn H-13 on @ 1000^{o}F
9. Check that steam is on by-pass; drain condensate from steam line and superheater by opening V-321 and V-327

10. Set FIC-115 (Stm) at 40 #/hr; turn H-14 on @ 1000°F
11. When PT-310 is at 99.5 psig, turn venturi scrubber pump on
12. Reset N_2 purges

Feed Hopper	10 SCFH
Feed Screw	100 SCFH
Reactor Shell	40 SCFH
Receiver	0 SCFH

13. Reset FIC-116 to 13.5 SCFM for remainder of heatup
14. When TT-201 reaches 400°F start coke feed (see coke feed instructions)
15. Reset FIC-115 (STM) to 15 #/hr
16. When TT-201 reaches 650°F start oxygen: reduce N_2 to 12.5 SCFM, set O_2 at 0.10 SCFM
17. If TT-201 does not respond within about 1 min., turn the oxygen off and let the bed heat to about 675°F before trying ignition again.
18. Bring TT-201 to 1400°F very slowly (having the bed temperature under control at this point tends to reduce problems during the remainder of the start-up)
19. When TT-201 reaches 1400°F, increase oxygen by 0.50 and take steam off bypass. (Note: It takes approximately 7 minutes to take steam off bypass -- allow plenty of time)
20. Bring TT-201 from 1400°F to 1800°F (or desired temperature) by increasing oxygen; during this time increase stm from 15 #/hr to the final value; decrease N_2 so as to maintain a space velocity of about 1.0
21. Set FIC-114 (stm) and FIC-116 (N_2) to their final values
22. Reset N_2 purges

Feed Hopper	0	
Feed Screw	100	SCFH
Reactor Shell	40	SCFH
Receiver	0	

23. When bed temperature is stable at desired temperature, set O_2 on cascade. While O_2 is on cascade, watch the bed temperature profile at all times. If the next higher thermocouple becomes hotter than the control thermocouple, remove O_2 from cascade control and lower the O_2 output by approximately 0.05. If this does not line out the temperature profile, reduce O_2 by 0.05 again. Once the temperature profile is correct, increase O_2 so as to bring the bed to the desired temperature. Return O_2 to cascade.

24. During start-up if the bed delta-P rises very high (>30) a sudden large (approximately 10 SCFM) increase in nitrogen input may loosen the bed and lower the ΔP.

COKE FEED INSTRUCTIONS

1. When TT-201 (01 on recorder) reaches 400°F start feed screw at 40 rpm (no removal)
2. Stop feed when 03 (TT-203, 25" bed) responds (approximately 2500 counts); Turn removal screw on and remove about 10 counts, then turn off
3. Do not feed or remove during remainder of heat-up
4. When O_2 is started set feed at 20 rpm and set removal at 20 rpm (this will very slowly build the bed; maintain these rates as steady as possible so as to aid the chief operator)
5. When the bed temperatures are near the final values and the bed ΔP is near normal slowly increase the feed RPM to the steady state value; maintain the removal rate at 20 rpm (this will build the bed)
6. When the bed level is established set the removal rate so as to maintain the bed at 48-50% of scale, on recorder.
7. If during start-up the bed lifts (indicated by TT-201 (or 01 on recorder) falling rapidly) turn feed screw off and set removal rate at about 10 rpm
8. At start of shutdown turn feed screw off and record both total feed counts and removal counts on Gasifier Report Sheet. After feed screw is shut off set removal at approximately 60-70 rpm.

GASIFIER SHUT-DOWN

1. Check that the propane to the flare is on
2. Turn off H-13 and H-14
3. Stop feed screw; record removal counts
4. Bring down O_2 flow while increasing N_2 to maintain U_o
5. Bring down Stm flow while increasing N_2; when steam is at approximately 20 #/hr put on bypass
6. Reset all AOV's

110	closed	101	open
111	closed	213	closed
115	closed	215	closed
116	open	241	closed
117	closed	254	closed
		270	open
		294	closed
		296	open
		297	closed

7. Set N_2 at approximately 10 SCFM and maintain until TT-201 is approximately $1200^{o}F$
8. Turn off scrubber pump and N_2
9. Close AOV-116 and AOV-270
10. Put PIC-310 in loop manual open valve to approximately 8%
11. Close AOV-101 and close purge N_2 valves; check shut-down of on-line instruments; turn off cyclone sample train; close shutter to nuclear level gauge; close main N_2 valve

AGRS OPERATION PROCEDURE

Day before Run

1. Turn dehydrators on; check N_2 purge flow (.5 SCFH); check that refrigerator oil heaters are on.
2. Complete pre-start-up checklist (see appendix)
3. For Syngas runs check for sufficient gas cylinder supply

Day of Run

3. Open refrigerator compressor isolation valves (3)
4. Reset alarm system at alarm panel
5. Set AOV's

AOV	Setting
309	Auto
322	Auto
340	Auto
356	Auto
397	Auto
399	Auto

AOV	Setting
305	Syngas auto
	integ. closed
333	auto
341	auto
360	closed
381	closed
393	auto
395	closed

AOV	Setting
310	auto
330	auto
345	auto
351	auto
352	auto
370	auto

AOV	Setting
131	auto
141	closed
151	closed
140	auto
150	auto

6. For a Syngas run check that PIC-310 is in auto and set at 100 psig
7. For a Syngas run set syngas N_2 at approximately 10 SCFM and set FIC-315 at approximately 6 SCFM; set PCV-345 (absorber) at 95 psig; open CO_2 tank valves

8. Set PIC-370 (stripper) in auto at 10 psig; set stripping N_2 at DV = 1
9. Using process N_2 (by opening V-248) set flash tank pressure to 30 psig using PIC-353
10. When absorber is at 95 psig reset FIC-315 to 4 SCFM
11. Check that LIC-341 and LIC-351 are in auto
12. When all pressures have stabilized turn on P-35 (solvent circulation pump) DO NOT set over 50%,when positive indication of solvent flow is seen (PT-354 pulsing) turn refrigerator system on and set TIC-349 at -35^{o}F; if P-35 is vapor locked close V-549 and momentarily open V-554. Immediately open V-549; repeat if necessary. CAUTION: Some solvent will be discharged down drain.
13. After indication of cooling is noted (TT-349 falling) begin to pressurize the absorber to 480 psig as follows;
    A. Set FIC-121 (N_2) at full open (15 psig output)
    B. Turn sourgas compressor (CP-31) on and close V-1006
    C. Reset PIC-345 (absorber) to 480 psig
    D. Reset FIC-315 to approximately 6 SCFM
    E. Reset syngas N_2 flow such that PIC-310 control valve is approximately 10-15% open
14. When absorber reaches about 200 psig reset PIC-353(flash tank) to 100 psig
15. When absorber bottoms temperature reaches 40^{o}F turn off process N_2 to flash tank (close V-248) and set PIC-353 to desire pressure
16. When temperatures reach the desired values initiate complete syngas flow using AGRS conditions report as a guide

INTEGRATED RUN PROCEDURE

Gasifier - use normal start-up

AGRS

1. Set PCV-308 at 100 psig
2. Open V-240 to supply start-up nitrogen to AGRS
3. Pressurize AGRS as normal (see AGRS operating procedure) but with FIC-315 less than 7 SCFM
4. Integration procedure
   A. Open V-552 (Gasifier-AGRS isolation valve)
   B. Set FIC-315 at approximately 1.0 SCFM
   C. Set PCV-308 such that PT-313 is approximately .6 psi lower than PT-310
   D. Place PIC-310 in loop manual
   E. Open AOV-305 (set in auto)
   F. Close V-240 (process N_2 supply)
   G. Remove PIC-310 from loop manual
   H. Increase FIC-315 slowly
5. Increase FIC-315 to desired value but not so high as to decrease PIC-310 valve position to less than 10%. Continue to monitor valve position of PIC-310 and if position becomes less than 4%, reduce setpoint of FIC-315

   Note: To conserve make-gas flow after integration open SX-2 and route gas to SX-1 O_2 monitor, then turn off SX-1 sample port. This will conserve approximately 0.7 SCFM of gas.

GASIFIER - AGRS SEPARATION PROCEDURE

1. If the oxygen monitoring point has been moved to SX-2 move it back to SX-1
2. Slowly lower FIC-315 to 1.0 SCFM (observe PCV-310 for erratic behavior)
3. Close AOV-305 and open V-240 at the same time
4. Set FIC-315 at 5 SCFM to purge system; increase stripping N_2 to DV = 3
5. Close V-552 (gasifier - AGRS isolation)

AGRS SHUT-DOWN

1. If run was a syngas run turn off flows of H_2S, mixed gas and CO_2; adjust N_2 flow to compensate
2. Set FIC-315 to 5 SCFM to purge system; set stripping N_2 DV = 3; reduce flash tank pressure to 100 psig and open V-248 (process N_2 supply) to set a flow of approximately 1.0 SCFM
3. Turn off refrigeration unit and valve off compressor
4. After all acid gases have been vented from the system turn solvent circulation pump P-35 off and allow solvent to drain from packing. After approximately 2 minutes place LIC-341 and LIC-351 in loop manual and close the valves
5. Close FCV-315 (in loop manual) and turn off CP-31 (sour gas compressor)
6. Turn syngas N_2 off (if syngas run) or close V-240 (if integrated run)
7. Turn off stripping N_2 to stripper; leave all pressure controllers in auto
8. Turn all AOV's to the closed position after manual power is restored. Audible click can be heard behind panel.
9. Turn all gas cylinders off
10. Turn off dehydrators D-31 (breaker #5)
11. If run was a syngas run and H_2S was used open AOV-310 (FIC-315 in loop manual; closed) and turn on AOV-140 and AOV-150 (purge N_2 supply to syngas box) leave these purges on for 5 min.; close AOV's 310, 140, 150
12. Close main N_2 valve

Appendix II

AGRS - Manual Valve Settings

I. Gas Lines

Valve	Position	Location	Level
V-237	open	N_2 supply to syngas	First floor
V-121	open	N_2 inlet to manifold	
V-122	open	N_2 inlet to manifold	
V-301	closed	CO_2 inlet to manifold	
V-303	closed	H_2S inlet to manifold	
V-305	closed	Mixed gas inlet to manifold	
V-307	closed	Manifold drain	
V-302	closed	Low point drain	
V-304	closed	Low point drain	
V-306	closed	Low point drain	
V-361	open	Manifold exit	
V-369	open	Syngas supply to dehydrators (closed for integrated run)	
V-546	closed	Gasifier - AGRS isolation valve (open for integrated run)	
V-552	open	Gasifier - AGRS isolation valve (closed for integrated run)	
V-240	closed	Process N_2 supply at dehydrators	
V-241	open	Purge N_2 to dehydrators	
V-368	closed	dehydrator bypass valve	
V-331	closed	F-33 drain	
V-264	open	Compressor isolation valve	
V-1006	open	Manual spill back valve	
V-1010	closed	Suction bottle drain	
V-1009	closed	Discharge bottle drain	
V-1013	closed	Knockout bottle drain	
V-1005	open	E-31 exit valve	
V-1014b	closed	Gas chiller by-pass	

Manual Valve Settings - Cont.

I. Gas Lines

Valve	Position	Location	Level
V-1015	open	Gas chiller exit	First floor
V-1023	open	Spill back access valve	
V-1024	closed	Surge bottle drain	
V-1025	closed	Compressor exit gas sampling valve	
V-1017	open	Purge N_2 to absorber bottom	
V-444	closed	Process N_2 to absorber bottom	
V-449	open	Purge N_2 to stripper bottom	
V-182	closed	Steam to reboiler	
V-158	closed	Steam exit from reboiler	
V-159	closed		
V-160	closed		
V-161	closed		
V-162	closed		
V-163	closed		
V-164	closed		
V-165	closed		
V-448	open	Purge N_2 to flash tank	Second floor
V-248	closed	Process N_2 to flash tank	
V-246	open	Gas exit from flash tank	
V-452	closed	Manual pressure release for flash tank	
V-1026	closed	Flash tank gas sample valve	
V-1027	closed	Absorber gas sample valve	Third floor
V-223	closed	Manual pressure release for absorber	
V-225	closed	Manual pressure release for stripper	
V-1028	closed	Stripper gas sampling valve	

Manual Valve Settings - Cont.

II. MeOH and Water Lines for MeOH system without trim heater

Valve	Position	Location	Level
V-587	open	MeOH supply to P-35	First floor
V-589	closed	Start-up tank isolation valve	
V-590	closed	MeOH supply to start-up pump	
V-548	closed	Drain line at P-35 exit	
V-554	closed	Drain line at P-35 exit	
V-549	open	P-35 exit	
V-363	closed	Start up pump exit	
V-1003	open	MeOH to gas chiller	
V-1035	closed	MeOH drain and vent line	
V-1004	closed	MeOH injection valve	
V-1016	closed	MeOH drain @ rupture plate	
V-1007	closed	MeOH drain and vent line	
V-335	open	MeOH restriction valve	
V-1001	closed	Block valve	
V-1002	closed	Block valve	
V-106	open	Cooling water supply to E-31	
V-1012	open	Cooling water discharge from E-31	
V-1011	closed	E-31 shell side vent	
V-1040	closed	Cooling water bypass for refrig.	
V-1041	open	Cooling water exit from refrig	
V-512	closed	MeOH bypass at refrig	
V-513	open	MeOH feed to refrig	
V-519	open	MeOH feed to refrig	
V-561	open	MeOH exit from refrig at E-36	Second floor
V-505	closed	At E-36	
V-506	closed		

Manual Valve Settings - Cont.

II. MeOH and Water Lines for MeOH system without trim heater

Valve	Position	Location	Level
V-507	closed		
V-508	closed		
V-509	closed		
V-510	closed		
V-511	closed		
V-484	closed		
V-485	closed		
V-1018	closed		
V-481	closed	Water supply at E-36	
V-482	closed		
V-154	closed		
V-1019	closed		
V-110	closed		
V-572	open only one	Top feed to absorber	Third Floor
V-573	open only one	Middle feed to absorber	Second Floor
V-555	open only one	Bottom feed to absorber	
V-526	open	MeOH bypass at F-31	
V-334	closed	MeOH sample tap at F-31	
V-525	closed	F-31 isolation	
V-527	closed	F-31 isolation	
V-536	closed	F-31 drain	
V-528	closed	F-31 drain	
V-550	closed	Absorber bottom drain	First floor
V-529	closed	Flash tank by pass	Second floor
V-450	open	Manual absorber level control	
V-533	closed	Flash tank drain	

Manual Valve Settings - Cont.

II. MeOH and Water Lines for MeOH system without trim heater

Valve	Position	Location	Level
V-530	open	Flash tank exit	Second floor
V-333	closed	MeOH sample tap at flash tank exit	
V-557	open	E-37 bypass	First floor
V-558	closed	E-37 bypass	
V-559	closed	E-37 bypass	
V-531	open	Feed to stripper	
V-534	closed	At E-37	
V-535	closed		
V-537	closed		
V-538	closed		
V-539	closed		
V-556	closed	Reboiler drain	
V-560	closed	Reboiler drain	
V-1024	open	Reboiler exit	
V-593	closed	E-37 bypass	
V-595	open	E-37 bypass	
V-594	closed	E-37 exit	
V-593	closed	E-37 exit	
V-520	closed	Chemical addition pump	
V-330	closed	MeOH sample tap at reboiler exit	
V-451	open	Manual level control for flash tank at stripper top	
V-112	closed	Cooling water for E-38	
V-157	closed	Cooling water for E-38	
V-156	closed	Cooling water for E-38	

MODIFICATION LIST FOR AGRS VALVING LIST

Using Lean/Rich Heat Exchanger

Liquid Side Valves

V-595	closed
V-593	open
V-594	open
V-557	closed
V-558	open
V-559	open

Integrated Run

Gas Side Valves

V-369	closed	syngas supply to dehydrators
V-546	open	Gasifier AGRS isolation valve
V-552	closed	Gasifier AGRS isolation valve

COAL GASIFICATION PLANT START UP CHECKLIST

Run No. _____ Date _____

Gasifier: Air _____ O_2 _____ Syngas _____ Integrated _____

Item	Setting or Reading	Initials	Date
1. Nitrogen tank level			
2. Nitrogen tank pressure			
3. a. Propane tank level			
b. Propane regulator pressure			
4. Main N_2 supply valve			
5. Main propane supply valve			
6. Main steam supply valve			
7. Main water supply valve			
8. Oxygen mainfold pressure			
9. Main 208 V breaker			
10. Main 440/480 V breaker			
11. Purge N_2 pressure PCV-123			
12. Process N_2 pressure PI-121			
13. Scrubber circulation pump Oil level and status			
14. Scrubber drain pump status			
15. Feed screw motor Oil level and status			
16. Removal screw motor Oil level and status			
17. Air compressor Oil level, belt tension, status			
18. Scrubber tank manway			
19. PCS Tank Cleaned			
20. Steam on by-pass			
21. O_2 regulator PCV-101 at 200 psi			
22. O_2 regulator PCV-110 at 135 psi			
23. Nuclear level gauge			
24. Feed hopper charged			
25. Feed hopper vent closed			
26. Feed coke sampled			
27. PCS tank level			
28. PCS valving configuration			

Item		Setting or Reading	Initials	Date
29.	V-324 closed			
	V-566A Open			
	V-566B Closed			
	V-501 Closed			
	V-583 Closed			
	V-567 Closed			
	V-564 Closed			
	V-581 Closed			
	V-565 Closed			
	V-546 Open			
	V-552 Closed			
30.	Reator pressure test	_____	_____	____
31.	Scrubber pump setting	_____	_____	____
32.	All unnecessary extension cords unpluged	_____	_____	____

ACID GAS REMOVAL SYSTEM START UP CHECKLIST

Run No._____ Date_____

Gasifier: Air_____ O_2_____ Syngas_____ Integrated_____

Item	Setting or Reading	Initials	Date
1. Refrigerator oil reservoir crankcase heaters on for 24 hrs			
2. Check manual valve settings (See Appendix A)			
3. a. Nitrogen tank level			
b. Nitrogen tank pressure			
4. a. Propane tank level			
b. Propane tank pressure			
5. Main N_2 supply valve			
6. Main propane supply valve			
7. Main water supply valve			
8. Purge N_2 pressure PCV 123			
9. Process N_2 pressure PI 122			
10. Check availability of gas cylinders (CO_2, H_2S, etc.) for syngas run			
11. Solvent circulation pump oil level and status			
12. Startup pump status			
13. Sour gas Compressor oil level, belt tension, status			
14. Syngas tank pressures			
a) CO_2 tank pressure			
, H_2S tank pressure			
c) Mixed Gas tank pressure			
15. Syngas supply pressure			
a) CO_2 supply pressure @ 250 psi			
b) H_2S supply pressure @ 125 psi			
c) Mixed gas supply pressure @ 135 psi			

Item		Setting or Reading	Initials	Date
16.	Dehydrators in line (V-368) closed			
17.	Purge N_2 to dehydrators set at .5 SCFM			
18.	Instrument air to dehydrators on			
19.	Drain suction, discharge and post-compressor surge bottles			
20.	Vent shellside of E-31 on sourgas compressor			
21.	Foxboro controllers settings			
	No. 1 set at 568 psig			
	No. 2 set at 50 psig			
22.	Manual spillback valve (V-1006) open			
23.	Valves on level sensing ΔP cell open			
24.	Level control valve for flash tank selected (back of rack 7)			
25.	Water supply to refrigerator open			
26.	Vent shellside of condenser on refrigerator			
27.	Refrigerant level in reservoir			
28.	Instrument air to refrigerator PI-350 set at 40 psig			
29.	Refrigerator crankcase oil level			
30.	Alarms for refrigerator (any on)			

Appendix III

GASIFIER - PCS - AGRS ALARMS

ALARM BUS CONTROL SWITCHES

POSITION	ACTION
UP:	Alarm, 2 Min. Delay, Shutdown
CENTER:	Alarm only
DOWN:	Immediate Shutdown (Oxygen, Air, and Steam off; Nitrogen full on. In the AGRS system, supply gasses and gas compressor off.)

TAG	CONDITION
FAL/H-114	Low/high oxidant feed rate; -10%,+10% fail to follow (FTF)
FAL/H-115	Low/high steam feed; +22%, -10% FTF
FAL/H-116	Low/high nitrogen feed; +10%, -10% FTF
TAH-130	Oxidant feed temp. > 1350°F
TAH-140	Steam feed temp. > 1557°F
PAL/H-114	Oxidant supply pres. < 110 or > 180 PSI
TAH-132	H-13 Heater temp > 2000°F
TAH-142	H-14 " " > 2000°F
DPAH/HH-201	Reactor differential press. > 25/> 48" Water
PAH-213	Reactor pressure > 110 PSI
DPAH-232	Scrubber diff. press > 98" Water
DPAH/L-231	Water injection differential Pressure < 5 or > 18 PSI
TAH-234	Scrubber exit temperature > 350°F

DPAH-291	Filter differential pressure > 30" Water
TAH-293	Sour gas temperature > 350°F
TAH-315	Absorber feed gas temp > 2% over set point
PAH-345	Absorber pressure > 540 PSI
TAL/H-349	Absorber solvent feed -5% FTF
TAL/H-390	Stripper column temperature <-20° or> 240°F
PAH-370	Stripper column pressure > 12.8 PSI
PAL/H-313	Compressor suction pressure <80 or> 120 PSI
PAL/H-315	Compressor discharge pressure < 200 or > 550 PSI
DPAH-315	Dehydrator differential pressure > 48" Water
DPAH-320	Gas chiller differential pressure > 48" Water
TAH-322	Sour gas temperature > 275°F
DPAH-340	Absorber differential pressure > 48" Water
PAL/H-353	Flash tank pressure < 75 or > 500 PSI
PAL/H-354	Solvent pump pressure < 350 or > 550 PSI
TAH-371	Vent temperature > 142°F
DPAH-390	Stripper differential pressure > 48" Water

The set points for the following switch-contact sensors are not available:

PAH-117	Feed gas pressure
LAL/H-252	Scrub water level
PAL-356	Refrigeration suction pressure
PAH-363	Refrigeration discharge pressure
TAH-363	Refrigeration discharge temperature
PAL-364	Refrigeration oil pressure
TAH-317	Compressor pressure

LAL/H-342	Absorber level
LAL/H-352	Flash tank level
LAL/H-392	Reboiler level
LAL-394	Reboiler coil level
PAL/H-122	Nitrogen pressure
PAL/H-131	Carbon dioxide supply pressure
PAH-141	Hydrogen sulfide supply pressure
PAH-151	Mixed gas pressure
PAH-160	Manifold pressure

The following alarms cause immediate pressure release and nitrogen purge:

TAH-201	Reactor temperature	> 2150°F
PAHH-213	Reactor pressure	> 125 psi
PAHH-345	Absorber pressure	> 570 psi
OXH-201	PCS oxygen high	
OXH-315	AGRS oxygen high	

The electric heaters H-13 and H-14, are turned off by alarms TAH-130, TAH-140, TAH-132, and TAH-142.

Appendix IV

Table 10

FACTORS FOR UNIT CONVERSIONS

Quantity	Equivalent Values
Mass	1 kg = 1000 g = 0.001 metric ton = 2.20462 lb_m = 35.27392 oz.
	1 lb_m = 16 oz. = 5 x 10^{-4} ton = 453.593 g = 0.453593 kg
Length	1 m = 100 cm = 1000 mm = 10^6 microns (μ) = 10^{10} angstroms (Å)
	= 39.37 in. = 3.2808 ft. = 1.0936 yards = 0.0006214 mile
	1 ft = 12 in. = 1/3 yd = 0.3048 m = 30.48 cm
Volume	1 m^3 = 1000 liters = 10^6 cm^3 = 10^6 ml
	= 35.3145 ft^3 = 220.83 imperial gallons = 264.17 gallons
	= 1056.68 quarts
	1 ft^3 = 1728 in^3 = 7.4805 gallons = 0.028317 m^3 = 28.317 liters
	= 28.317 cm^3
Force	1 N = 1 kg · m/s^2 = 10^5 dynes = 10^5 g · cm/s^2 = 0.22481 lb_f
	1 lb_f = 32.174 lb_m · ft/s^2 = 4.4482 N = 4.4482 x 10^5 dynes
Pressure	1 atm = 1.01325 x 10^5 N/m^2 (Pa) = 1.01325 bars
	= 1.01325 x 10^6 dynes/cm^2
	= 760 mm Hg @ 0°C (torr) = 10.333 m H_2O @ 4°C
	= 14.696 lb_f/in^2 (psi) = 33.9 ft H_2O @ 4°C
	= 29.921 in Hg @ 0°C
Energy	1 J = 1 N · m = 10^7 ergs = 10^7 dyne · cm
	= 2.778 x 10^{-7} kW · hr = 0.23901 cal
	= 0.7376 ft-lb_f = 9.486 x 10^{-4} Btu
Power	1 W = 1 J/s = 0.23901 cal/s = 0.7376 ft · lb_f/s = 9.486 x 10^{-4} Btu/s
	= 1.341 x 10^{-3} hp